Research Article

Synthesis of 2,3-dihydroquinazolinones using nano-ovalbumin as a non-toxic biocatalyst



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Abstract

Bio-catalysts are non-toxic, metal-free, abundant, and efficient materials with less waste generation that decrease the environmental pollution. Nano-ovalbumin is a biodegradable phosphoglyco protein with isoelectric point (pl) of 4.5, molecular weight of 44.5 kDa containing 385 residues of amino acids. Nano-ovalbumin can be used as a non-toxic heterogeneous and eco-friendly catalyst for promotion of organic compounds. Quinazolinones have many biological and medicinal activities such as anticonvulsant, antitumor, anticancer, hypnotic/sedatives, antimalarial, antibacterial, antioxidant, antimicrobial, antifungal, cytotoxic, antiproliferative, and inhibitory activities on α -glucosidase. In this research, nano-ovalbumin was ready by a new, easy, inexpensive and convenient protocol from egg white and used as a heterogeneous biocatalyst in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones via two-component reaction of 2-aminobenzamide with aldehyde or ketone. This eco-friendly catalyst carried out the reaction in green conditions and with good to excellent yields. The structure of products were determined by Fourier Transform-Infrared (FT-IR), ¹H-Nuclear Magnetic Resonance (¹H-NMR) and their physical properties were compared with those reported in others work.

Keywords Nano-biocatalyst · 2,3-Dihydroquinazolinone · Eco-friendly catalyst · Nano-ovalbumin

1 Introduction

2,3-Dihydroquinazolin-4(1*H*)-ones are a significant class of heterocyclic compounds that form vary useful moiety of pharmaceutical, agricultural chemical and veterinary products [1, 2]. Quinazolinone and its derivatives have many biological and medicinal activities such as anticonvulsant [3], antitumor [4], anticancer [5], hypnotic/sedatives [6], antimalarial [7], antibacterial and antioxidant [8], antimicrobial [9], antifungal and cytotoxic [10], antiproliferative [11], and inhibitory activities on α-glucosidase [12]. Several examples of marketed drugs with the 2,3-dihydroquinozolinones (DHQ) framework are shown in Fig. 1. Several methods have been published for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones including the two-component reaction of 2-aminobenzamide with aldehyde or ketone and the three-component reaction of isatoic anhydride, aldehydes, and amines, or ammonium acetate [13].

This reaction has been catalyzed by different catalysts including (MNPs-PSA) [14], Wang-OSO₃H [15], 2-morpholinoethanesulfonic acid [16], nanometasilica disulfuric acid (NMSDSA) [17], CuCl₂/Fe₃O₄-TEDETA [18], [Bmim]PF₆ [19], nano-Fe₃O₄/TiCl₂/cellulose [20], tetrabutylammoniumbromide (TBAB) [21], ammonium chloride [22], NH₂SO₃H and MCM-41@serine@Cu(II) [23]. However, some of these methods have disadvantages such as the use of costly reagents or toxic organic solvents, difficulty in preparation

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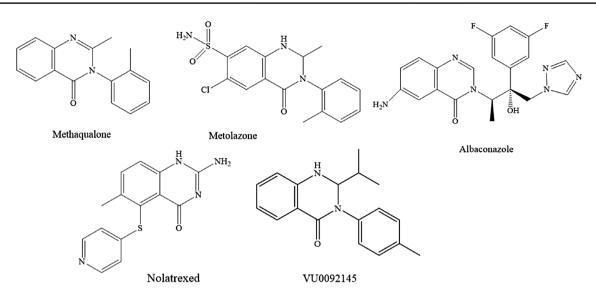


Fig. 1 Some important DHQs in marketed drugs

and/or storage of catalysts, tedious work-up and separation of catalyst from the products.

In recent decades, application of biocatalysts in organic synthesis has attracted more attention. Bio-catalysts are non-toxic, metal-free, abundant, and efficient materials with less waste generation that decrease the environmental pollution. Albumin as the major component of egg white is a phosphoglyco protein that has attracted considerable attention from the early twentieth-century. 9.7–12% of egg white is protein that 54% of it is ovalbumin. The isoelectric point (pl) of this protein is 4.5 and it contains 385 residues of amino acids with the molecular weight of 44.5 kDa [24].

Several methods have been proposed for the purification of this proteins, including gel penetration and anion exchange chromatography [25], Q Sepharose fast flow column [26], using polyethylene glycol precipitation and isoelectric precipitation [27]. In continuation of our works about application of heterogeneous catalysts in organic synthesis [28–30], In this research, nano-ovalbumin was ready by a new, easy, inexpensive and convenient protocol from egg white [31, 32] and used as a heterogeneous biocatalyst in the synthesis of 2,3-dihydroquinazolinones.

2 Experimental materials and methods

The chemical compounds were acquired from Merck, Aldrich, and Fluka chemical companies and were used without additional purification. 2-Aminobenzamide was prepared from Merck Company. All used aldehydes were previously prepared from Merck, Aldrich or Fluka Companies. Nano-ovalbumin was prepared from egg white

SN Applied Sciences A Springer Nature journal according to reported procedure from our research team [31, 32]. The products were determined by FT-IR, 1H-NMR and their physical properties were compared with those reported in the other work. Fourier transform infrared (FT-IR) spectra were run on a Bruker, Equinox 55 spectrometer. A Bruker (DRX-400 Avance) nuclear magnetic resonance (NMR) was used to record the ¹H-NMR spectra. Melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus and were uncorrected.

2.1 General procedure for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones in the presence of nano-ovalbumin

In a 25-mL round bottom flask, nano-ovalbumin (0.2 g) was added to a solution of 2-aminobenzamide (1 mmol) and aldehyde (1.25 mmol) in 5 mL of EtOH: H_2O (2:1) and the mixture was refluxed for an appropriate time. After monitoring the reaction by TLC (Ethyl acetate: *n*-Hexane, 1:4) and completing, ethanol (5 mL) was added to the mixture for separating the catalyst from the product. Then, water (10 mL) was added to the filtrate and the solid products were appeared. The obtained products were filtered and washed with water (2×5 mL) and dried at room temperature.

3 Results and Discussion

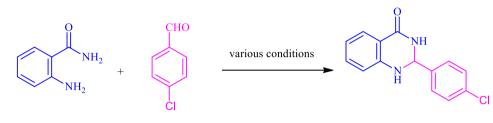
The catalytic activity of nano-ovalbumin was evaluated in the synthesis of 2,3-dihydroquinazolin-4(1H)-ones under different conditions. For optimization of the conditions, the model reaction between 2-aminobenzamide (anthranilamide; 1 mmol) and 4-chlorobenzaldehyde (1.25 mmol) was selected. The best condition was using 0.2 g of nano-ovalbumin under reflux (Ethanol/H₂O (2:1)) condition (Table 1, entry 12).

The catalytic activity of nano-ovalbumin was investigated in the synthesis of 2,3-dihydroquinazolin-4(1*H*)ones by the reaction of various types of aldehydes with anthranilamide (Table 2). Checking different aldehydes with electron-donating or electron-withdrawing groups under optimized conditions showed good reactivity of the catalyst due to high yields without any side products (Table 2, entries 2–11).

In addition of aldehydes, we have also applied ketones for production of 2,3-dihydroquinazolin-4(1*H*)-ones. If we have applied cyclic ketones such as cyclohexanone or cyclopentanone, the product 2,3-dihydroquinazolin-4(1*H*)ones with spiro skeletone have been formed (Scheme 1, Table 3).

A presumed mechanism for the preparation of 2,3-dihydroquinazolin-4(1H)-one corresponding to the

Table 1 Optimization of the reaction conditions for the synthesis of 2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one.



Ent	Solvent	Catalyst (g)	Conditions	Time (h)	Yield (%) ^a	Ref
1	_	Nano-ovalbumin (0.2)	r.t	2	_	_
2	-	Nano-ovalbumin (0.2)	80 °C	2	30	-
3	EtOH	Nano-ovalbumin (0.1)	Ultrasound	0.5	_	-
4	_	Nano-ovalbumin (0.1)	Ball-mill	0.6	_	-
5	_	Nano-ovalbumin (0.1)	Microwave	0.6	80	-
6	PEG-400	Nano-ovalbumin (0.1)	Microwave	0.75	85	-
7	PEG-400	Nano-ovalbumin (0.1)	100 °C	5	50	-
8	_	Nano-ovalbumin (0.1)	Grinding	0.75	50	-
9	H ₂ O	Nano-ovalbumin (0.2)	Reflux	3.5	50	-
10	EtOH	Nano-ovalbumin (0.2)	Reflux	3.5	85	-
11	EtOH:H ₂ O ^b	Nano-ovalbumin (0.2)	Reflux	3.5	92	-
12	EtOH:H ₂ O ^c	Nano-ovalbumin (0.2)	Reflux	1.5	96	-
13	EtOH:H ₂ O ^c	Nano-ovalbumin (0.1)	Reflux	3	78	-
14	EtOH:H ₂ O ^c	Nano-ovalbumin (0.05)	Reflux	5	65	-
15	EtOH:H ₂ O ^c	_	Reflux	3	50	-
16	EtOH	_	Reflux	3	45	
17	H ₂ O	_	Reflux	3	30	
18	H ₂ O	MNPs-PSA ^d	70 °C	0.4	90	[14]
19	EtOH:H ₂ O	MES ^e	60 °C	2	89	[16]
20	EtOH	CuCl ₂ /Fe ₃ O ₄	Reflux	0.75	98	[18]
21	_	[Bmim]PF ₆	75 °C	0.6	90	[19]
22	_	TBAB ^f	100 °C	1	75	[21]
23	H ₂ O	NH ₂ SO ₃ H	70 °C	0.58	89	[23]
24	H ₂ O	Wang-OSO ₃ H	100 °C	0.4	84	[15]

^alsolated yield

^bRatio 1:1

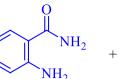
^cRatio 2:1

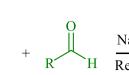
^d*N*-Propylsulfamic acid supported on magnetic Fe₃O₄ nanoparticles

^e2-Morpholinoethanesulfonic acid

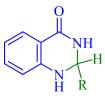
^fTetrabutylammonium bromide

Table 2 Synthesis of 2,3-dihydroquinazolin-4(1H)-ones catalyzed by nano-ovalbumin



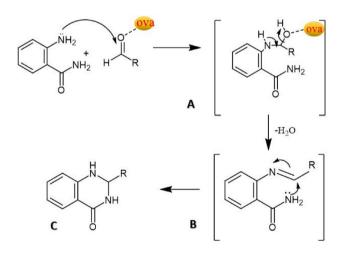


Nano-ovalbumin Reflux EtOH/H₂O



Entry	R	Time (h)	Yield (%) ^a	M.P. (°C) Found	M.P. (°C) Reported [Ref.]
1	Ph	0.75	89	208–210	216–218 [22]
2	4-NO ₂ -Ph	1.5	94	202–207	200–202 [33]
3	4-Cl-Ph	1.5	96	214–216	214–216 [<mark>34</mark>]
4	4-Isopropyl-Ph	3.5	67	155–160	158–161 [<mark>20</mark>]
5	3-NO ₂ -Ph	1.5	89	190–193	193–195 [<mark>20</mark>]
6	2-NO ₂ -Ph	2	87	190–192	191–192 [<mark>35</mark>]
7	2-Cl-Ph	1	90	207–209	206–208 [<mark>36</mark>]
8	2,4-Cl ₂ -Ph	2	88	167–170	166–169 [<mark>20</mark>]
9	2,4-(MeO) ₂ -Ph	3	67	185–187	185–187 [<mark>37</mark>]
10	3-OMe-4-OH	1.25	60	225-230	238–240 [<mark>38</mark>]
11	4-(Me ₂ N)-Ph	1	84	197–202	208–210 [<mark>16</mark>]
12	<i>n</i> -Pentyl	1.5	77	152–155	154 [<mark>39</mark>]
13	Cyclohexyl	1	88	173–176	174–175 [<mark>40</mark>]

Reaction conditions: 2-aminobenzamide (1.0 mmol), aldehyde (1.25 mmol) and nano-ovalbumin (0.2 g) ^alsolated yield



Scheme 1 Presumed mechanism of the cyclocondensation of anthranilamide and an aldehyde

condensation of anthranilamide and an aldehyde in the presence of nano-ovalbumin was shown in scheme. Initially, the nucleophilic attack of the nitrogen of the amino group of the anthranilamide on the carbonyl carbon of the aldehyde, promoted by the catalyst, results in the formation of hydroxyl intermediate **A**. Next, the catalyst promotes the formation of the Schiff base **B** from **A** through

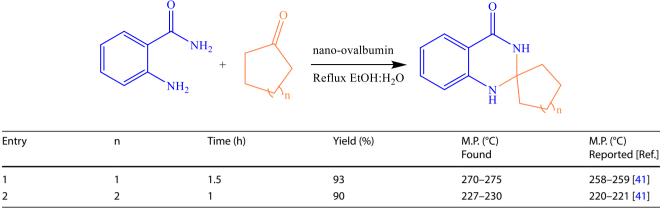
SN Applied Sciences A Springer Nature journal the removal of a water molecule. Finally, the imine undergoes intramolecular cyclization by nucleophilic attack of the nitrogen of the amide group on the imine carbon to furnish the corresponding final product **C**.

The reusability of the catalyst as an important issue was also investigated. Nano-ovalbumin was recovered conveniently and efficiently from the reaction mixture by filtration, washing with water (5 mL) and acetone (5 mL) and then drying at room temperature. It was observed that the recovered biocatalyst could be used at four times without significant loss of activity (Fig. 2).

4 Conclusion

An efficient and environmentally benign method has been developed for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones in the presence of a catalytic amount of nano-ovalbumin in ethanol/H₂O under reflux condition. Egg white nano-ovalbumin as an environmentally benign biocatalyst was prepared from egg white by a simple protocol. Simple separation of catalyst, reusability of eco-friendly metal-free biocatalyst and excellent yields are advantages of this protocol.

Table 3 Synthesis of spiro-2,3-dihydroquinazolin-4(1H)-ones catalyzed by nano-ovalbumin



Reaction conditions: 2-aminobenzamide (1.0 mmol), ketone (1.25 mmol) and nano-ovalbumin (0.2 g)

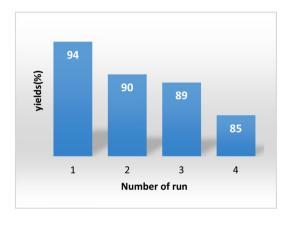


Fig. 2 Recyclability of the catalyst

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Compliance with ethical standards

Conflict of interestConflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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