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Application of SBA-Pr-SO₃H in the synthesis of 2,3-dihydroquinazoline-4(1*H*)-ones: characterization, UV–Vis investigations and DFT studies

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Abstract An efficient one-pot synthesis of 2,3-dihydroquinazoline-4(1*H*)-one derivatives **4a–l** is described using SBA-Pr-SO₃H as a heterogeneous acid catalyst. The present methodology resulted in various derivatives of 2,3-dihydroquinazoline-4(1*H*)-one in good yield via a three-component reaction of isatoic anhydride, aldehydes and ammonium acetate. SBA-Pr-SO₃H played a significant role as an efficient mesoporous catalyst due to its pore size of 6 nm. Additionally, UV–Vis spectrum of the products was studied in order to investigate their application as UV absorbers.

Keywords SBA-Pr-SO₃H \cdot 2,3-Dihydroquinazoline-4(1*H*)-one \cdot Multicomponent reaction \cdot Isatoic anhydride \cdot UV absorbers

Introduction

2,3-Dihydroquinazoline-4(1H)-ones are important heterocyclic compounds with a broad range of biological and pharmacological properties including antitumor, antihistamine, analgesic, anticancer, vasodilating and diuretic activities [1–4]. For example, febrifugine, as a quinazolinone

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alkaloid with antimalarial properties extracted from *Dichroa febrifuga*, was firstly synthesized by McLaughlin and coworkers (Fig. 1) [3]. In the view of their importance and useful properties, several procedures for preparing of 2,3-dihydroquinazolin-4(1*H*)-ones have been reported in the past few years, such as: (1) the reaction of aldehydes or ketones with anthranilamide using Lewis or Brønsted acids as the catalyst [5, 6]; (2) the reductive cyclization of 2-nitrobenzamide with different aldehydes or ketones by the use of SnCl₂ as the catalyst [7]; (3) the reaction of primary amine (or ammonium salts) and isatoic anhydride, with aldehydes or ketones using Lewis or Brønsted acids as the catalyst [8–10].

Natural or artificial ultraviolet (UV) light is responsible for the destruction and decomposition of organic compounds, the dyes discoloration and the loss of mechanical properties in polymeric compounds and plastics [11]. Many efforts have been made to improve the durability of these materials upon exposure to UV light. The use of organic UV absorbers in the matrices of the mentioned materials is one of the most convenient and effective methods to avoid their photoaging [12]. The organic UV absorbers must, of necessity, be colorless compounds and absorb UV light in the range of 280 and 400 nm [13].

SBA-15, as a mesoporous silica, was synthesized by Zhao and coworkers for the first time in 1998 [14]. The internal surface of SBA-15 can be functionalized with different organic functional groups [15–18] to produce an effective and active catalyst. It has exceptional advantages such as high surface area, large pore size and high thermal stability; in conclusion, it shows excellent catalytic activity in organic transformations [19–21]. In this paper, sulfonic acid functionalized silica (SBA-Pr-SO₃H) was used as a heterogeneous Brønsted acid catalyst in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives. Then, in

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order to investigate their application as UV absorbers, the UV–Vis spectrum of the products was studied.

Experimental

Materials and techniques

Fourier transform infrared (FT-IR) spectra were recorded on KBr disks using a FT-IR Bruker Tensor 27 instrument. Melting points were measured by the capillary tube method using an electro thermal 9200 apparatus. The ¹H NMR (250 MHz) and ¹³C NMR (62.5 MHz) spectra were run on a Bruker DPX using tetramethylsilane (TMS) as an internal standard in CDCl₃ and/or DMSO-d₆ solution. Mass spectrometry (MS) analysis was performed on a model 5973 mass-selective detector (Agilent). UV–Vis spectrum was run on an Analytik Jena Specord[®] S600 spectrophotometer.

Quantum chemical calculations

All of the quantum chemical calculations were accomplished using DFT method at B3LYP/6-31g(d) level by Gaussian 98 program package. The optimized structures **4a–I** were subjected to some quantum chemical parameters such as the energy of the highest occupied molecular orbital (E_{HOMO}), the energy of the lowest unoccupied molecular orbital (E_{LUMO}) and the energy gap between these orbitals ($\Delta E = E_{\text{HOMO}} - E_{\text{LUMO}}$).

Production and functionalization of SBA-15

The nanoporous compound SBA-15 was synthesized and functionalized according to our previous report [22, 23], and the modified SBA-Pr-SO₃H was used as a mesoporous solid acid catalyst in the following reaction.

General procedure for the preparation of quinazolinone compounds 4a–l

The SBA-Pr-SO₃H (0.02 g) was firstly activated under vacuum and then after cooling to room temperature, isatoic

anhydride 1 (1 mmol, 0.163 g), aromatic aldehyde 2a–l (1 mmol) and ammonium acetate 3 (1.2 mmol) were added to it. The mixture was heated under solvent-free condition at 115 °C for an appropriate time. Completion of the reaction was monitored through thin layer chromatography technique (TLC). The resulting solid product was dissolved in hot EtOH and filtered to remove the unsolvable catalyst, and then the filtrate was cooled to give the pure product. The spectroscopic and analytical data for the new products are presented in the following part.

Spectral data for some compounds

2-(2,6-dichlorophenyl)quinazolin-4(3H)-one (4j) FT-IR (KBr): υ (cm⁻¹) = 3433, 3189 (NH), 3062 (C–H Aromatic), 1650 (C=O), 1517, 1436 (C–C Aromatic). ¹H NMR (250 MHz, DMSO- d_6): $\delta_H = 6.61$ (t, J = 7.5 Hz, 1H, Ar–H), 6.75 (s, 1H, Ar–H), 7 (s, 1H, Ar–H), 7.9 (t, J = 6.25, 1H, Ar–H), 7.3–7.7 (m, 3H, Ar–H), 8.3 (s, 1H, NH) ppm. ¹³C NMR (62.5 MHz, DMSO- d_6): $\delta_C = 64.9$, 113.79, 114, 116.9, 127.6, 130.1, 131.4, 133.7, 133.9, 135.8, 148.2, 163.2 ppm. MS (m/e): 292 (M⁺, 18%), 120 (68%), 92 (38%), 65 (17%).

2,3-dihydro-2-(3-methoxyphenyl)quinazolin-4(1*H***)-one** (**4k**) FT-IR (KBr): υ (cm⁻¹) = 3291, 3190 (NH), 3051 (C–H Aromatic), 1518, 1490 (C–C Aromatic), 1647 (C=O), 1149 (C–O). ¹H NMR (250 MHz, CDCl₃): $\delta_{\rm H} = 3.82$ (s, 3H, CH), 5.86 (s, 1H, NH), 6 (s, 1H, C–H), 6.68 (d, *J* = 8, 1H, Ar–H), 6.92 (m, 2H, Ar–H), 7.12 (*t*, *J* = 7.5, 2H, Ar–H), 7.33 (*m*, 2H, Ar–H), 7.91 (*d*, *J* = 25, 1H, Ar–H), 7.94 (*s*, 1H. NH) ppm. ¹³C NMR (62.5 MHz, CDCl₃): $\delta_{\rm C} = 55.3$, 68.9, 112.3, 114.5, 115.9, 119.6, 126.3, 128.6, 130.1, 134.7 ppm. MS (m/e): 254 (M⁺, 65%), 147 (100%), 120 (70%), 92 (43%), 77 (13%), 65 (20%).

2,3-dihydro 2-(5-methylfuran-2-yl)- quinazolin-4(1*H***)one (4l)** FT-IR (KBr): v (cm⁻¹) = 3267, 3187 (NH), 3067 (C–H Aromatic), 2921 (C–H Aliphatic),





Scheme 1 Synthesis of 2,3-dihydroquinazolin-4(1H)-one 4a-l

Table 1 The optimization of reaction conditions for the synthesis of2,3-dihydroquinazolin-4(1H)-one

Entry	Solvent	Catalyst	Tempera- ture (°C)	Time (h)	Yield (%)
1	CH ₃ CH ₂ OH	SBA-Pr- SO ₃ H	Reflux	7 h	70
2	H ₂ O	SBA-Pr- SO ₃ H	Reflux	12 h	36
3	CH ₃ CN	SBA-Pr- SO ₃ H	Reflux	3 h	50
4	Neat	SBA-Pr- SO ₃ H	120 °C	4 min	90
5	Neat	_	120 °C	5 h	40

1645.73 (C=O). ¹H NMR (250 MHz, DMSO-*d*₆): $\delta_{\rm H} = 2.2$ (*s*, 3H, CH Aliphatic), 5.66 (*s*, 1H, NH), 5.9 (*s*, 1H, Ar–H), 6.0 (*s*, 1H, Ar–H), 6.7 (td, 2H, *J* = 3), 7.1 (*s*, 2H, Ar–H), 7.4–7.8 (*m*, 1H, Ar–H), 8.3 (*s*, 1H, NH) ppm. ¹³C NMR (62.5 MHz, DMSO-*d*₆): $\delta_{\rm C} = 13.7$, 60.7, 106.7, 108.5, 114.9, 117.6, 126.6, 127.7, 133.6, 147.6, 151.8, 152.9, 163.7 ppm. MS (m/e): 226 (M⁺⁺, 100%), 211(42%), 147 (15%), 120 (46%), 92 (21%).

Results and discussion

Synthesis of 2,3-dihydroquinazoline-4(1*H*)-ones was performed through the one-pot three-component condensation of isatoic anhydride **1**, aromatic aldehydes **2a–1** and ammonium acetate **3** in the presence of SBA-Pr-SO₃H (Scheme 1). To optimize the reaction conditions for compound **4a** (Ar=Ph), different solvents were studied including H₂O, EtOH, CH₃CN as well as solvent-free systems (Table 1). Among the tested conditions, the use of SBA-Pr-SO₃H (0.02 g) under solvent-free system showed the best results with excellent product yield and shortest reaction time (4 min).

Additionally, the amount of SBA-Pr-SO₃H was modified for the synthesis of 2,3-dihydroquinazolin-4(1*H*)one **4a** as shown in Table 2. The results confirmed that by increasing the catalyst amount from 0.02 to 0.04, the

Table 2 The effect of catalyst amount on the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one 4a

Entry	Catalyst (g)	Time (min)	Yield (%)
1	0.01	5	66
2	0.02	4	90
3	0.03	4	87
4	0.04	4	88

reaction yield does not change significantly. Therefore, 0.02 g of catalyst was selected as modified amount for the further reactions.

Therefore, this reaction was developed with different aldehydes under solvent-free condition and the results have been summarized in Table 3. After completion of the reaction (monitored by TLC), the crude product was dissolved in hot EtOH, and the catalyst was easily filtrated from the reaction mixture. The acid catalyst can be reactivated by washing with hot EtOH and subsequently with a dilute acid solution, water and acetone to reuse for several times without significant loss of activity. The new products were characterized by melting point, FT-IR, GC–MS and NMR spectral data. Melting points of the known products were compared with the reported values in literature as displayed in Table 3.

In order to study the effect of physical characteristics of SBA-Pr-SO₃H as catalyst in this reaction, the model reaction was tested in the presence of sulfonic acid modified amorphous silica (SiO₂-Pr-SO₃H). As illustrated in Table 3, Entries 1–2, the reaction time and the product yield were significantly increased and decreased, respectively, in the presence of SiO₂-Pr-SO₃H compared to SBA-Pr-SO₃H. Since the substrate constituent of both these catalysts is same, it can be, therefore, concluded that the mesoporous structure of SBA-Pr-SO₃H provides appropriate space for treating the reactants to gain the product. Additionally, the conditions of present methodology were compared with some other published methods and are summarized in Table 4 (Entries 3-5). As it is clear, the safe and green SBA-Pr-SO₃H catalyzed and accelerated the reaction more efficiently to give the higher yield of product within shorter reaction time.

Table 3Synthesis of2,3-dihydroquinazoline-4(1H)-ones 4a-l in the presence ofSBA-Pr-SO₃H as catalyst

Entry	No.	Ar	Time (min)	Yield (%)	Crystal colors	m.p. (°C)	m.p. (Lit)
1	4a	4-OMe-C ₆ H ₄	4	90	White	190–192	192–193 [24]
2	4b	4-Me-C ₆ H ₄	7	87	White	229-231	229–231 [<mark>25</mark>]
3	4c	$4-OH-C_6H_4$	5	93	White	285-287	285–287 [<mark>26</mark>]
4	4d	2,3-Cl ₂ C ₆ H ₃	5	90	Cream	232-233	233–235 [25, 26]
5	4e	4-Cl-C ₆ H ₄	5	97	White	206-208	207–208 [25, 26]
6	4f	2-OMe-C ₆ H ₄	25	78	White	173–175	173–175 [25, 26]
7	4g	$4-F-C_6H_4$	10	88	White	204-206	205–207 [27]
8	4h	2,4-(OCH ₃) ₂ C ₆ H ₃	8	87	White	186–188	186–187 [<mark>27</mark>]
9	4i	$2-NO_2-C_6H_4$	30	92	Light orange	190–193	190–193 [27]
10	4j	2,6-Cl ₂ C ₆ H ₃	40	90	Cream	167-170	New
11	4k	3-OMe-C ₆ H ₄	10	99	White	148-150	New
12	41		30	65	Brown	173–175	New

Table 4 Comparing
the reaction conditions
in the synthesis of
2,3-dihydroquinazolin-4(1H)-
one 4a

Entry	Solvent	Catalyst	Conditions	Time (min)	Yield (%)	Year [ref.]
1	Neat	SBA-Pr-SO ₃ H	120 °C	4	90	This work
2	Neat	SiO ₂ -Pr-SO ₃ H	120 °C	7	77	This work
3	EtOH	Ga(OTf) ₃	Reflux	40	83	2008
4	EtOH	I ₂	Reflux	70	89	2009
5	Neat	Al/Al ₂ O ₃ nanoparticles	120 °C	8	80	2010



Scheme 2 A proposed mechanism for the formation of 4a-l

The reaction mechanism of quinazolinone compounds is similar to that of Friedländer reaction [28]. Previously, Javanshir and coworkers investigated the effect of p-toluenesulfonic acid as catalyst in the reaction mechanism of quinoline compounds through DFT computational method [29]. Correspondingly, the proposed mechanism for the formation of compounds **4a–l** can be explained as shown in Scheme 2. The carbonyl group of isatoic anhydride **1** is firstly protonated



Fig. 2 Synthesis of 2,3-dihydroquinazolin-4(1H)-one derivatives using SBA-Pr-SO₃H as a nanoreactor

by the solid acid catalyst to give intermediate **5**. Then, this intermediate can be easily attacked by ammonia from the ammonium acetate **3**, followed by decarboxylation to produce 2-aminobenzamide **6**. Condensation of the protonated aromatic aldehyde with 2-aminobenzamide **6**, followed by the loss of a water molecule gives imine **8**, which then undergoes an intramolecular cyclization to afford the desired product **4a–l** (Scheme 2). SBA-Pr-SO₃H can act as a nanoreactor where the reaction takes place easily in its nanopores (Fig. 2).

The catalyst recyclability was also examined under optimized conditions for the synthesis of compound **4a**. In this regard, the reaction was performed in the first cycle for four times to recover about 0.06 gr of SBA-Pr-SO₃H. Subsequently, it was washed with hot EtOH and reactivated using diluted H_2SO_4 and then reused. Then, the recycling process was repeated for four times and the product yields were 90, 84, 83 and 81%. It was found that the catalytic activity drops slightly from the first cycle to the second cycle due to leaching of some sulfonic acid groups which were not grafted well on the surface of SBA-15. Additionally, no significant drop of catalytic activity was detected for the third and fourth cycles which means the catalyst is stable after leaching the week grafted organic groups. In fact, this catalyst is completely recoverable.

Investigation of UV-Vis spectrum

The UV spectra of 2,3-dihydroquinazolin-4(1H)-one derivatives **4a–1** were obtained in ethanol at 5×10^{-5} M concentration (Fig. 4). The results showed that a number of 2,3-dihydroquinazolin-4(1H)-one derivatives may have potential as UV absorbers. While all compounds displayed very strong absorptions in the UV-A region (239-275 nm), the most interesting are those compounds which can absorb in the range of 280 and 400 nm (UV-B and UV-A regions). Compound 4c showed an excellent absorption at UV-B, while the absorptions of 4a, 4b, 4e and 4j were in a range of UV-A. Among the compounds, 4d, 4f, 4g, 4l and 4k have a broad range absorption from 260 to 400 nm; the absorption of **4k** was so strong. Therefore, these compounds are good candidates as UV absorbers, although compounds 4h and 4i showed a weak absorption.

In order to have a better insight on the electronic properties, the quantum chemical calculations of the compounds **4a–l** were performed by density functional theory (DFT) to determine the HOMO and LUMO. We choose a B3LYP/6-31G(d) level for DFT calculations. The density distributions of HOMO and LUMO orbitals for the product **4a** with optimized geometry are presented in Fig. 3a and b, respectively. According to Fig. 3a–b, it is obvious that the HOMO electron density is distributed on the whole atoms of quinazoline ring; however, in LUMO structure, the density is diffused on the ring part. Therefore, the quinazoline ring (in the ground state of the molecules) is the active cite for the absorption of the photons from the UV irradiation.

Herein, the quantum chemical parameters such as the energy of the HOMO and LUMO orbitals and the band gap energy of **4a–1** were calculated by DFT and then compared to the calculated band gap energy and ΔE from the λ_{max} (Table 5). The λ_{max} was obtained from the UV–Vis spectra of each compounds in EtOH (Fig. 4). The data obtained by DFT are in agreement with the experimental data (UV–Vis) except compound **4e**. Since the transition energies of HOMO \rightarrow LUMO (S₁ \leftarrow S₀) are in the range of UV absorber compounds, it means that the synthesized compounds **4a–1** may be used as UV absorbers.

Fig. 3 Frontier molecular orbital density distributions of compound 4a



Entry	Compound no.	Calculated data using DFT method				Obtained data via UV–Vis spectrum	
		λ_{\max} (nm)	Oscillator strength (f)	Predicted HOMO-LUMO gap (ev)	λ_{\max} (nm)	HOMO–LUMO gap (ev) from $E = hc/\lambda_{max}$	
1	4 a	313	0.0417	3.96	340	3.6	
2	4b	312	0.0436	3.98	345	3.6	
3	4c	314	0.0415	3.95	300	4.1	
4	4d	336	0.0026	3.69	270	4.5	
5	4e	317	0.0319	3.92	345	3.6	
6	4f	325	0.0357	3.82	318	3.9	
7	4g	315	0.037	3.94	320	3.8	
8	4h	353	0.004	3.52	350	3.5	
9	4i	325	0.0412	3.82	345	3.6	
10	4j	587	0.0141	2.11	350	3.5	
11	4k	313	0.0394	3.96	345	3.6	
12	41	355	0.2374	3.50	330	3.7	

 Table 5
 The quantum chemical parameters derived from compounds 4a-l using DFT method and obtained by UV-Vis spectrum



Fig. 4 UV-Vis spectrum of 2,3-dihydroquinazolin-4(1H)-one derivatives 4a-l in EtOH

Antimicrobial and antifungal tests

All products **4a-1** were investigated for antimicrobial and antifungal activities using disk diffusion method. The

microorganisms used in this study were *Escherichia coli* and *Pseudomonas aeruginusa* as gram negative bacteria, *Bacillus subtilis* and *Staphylococcus aureus* as gram positive bacteria and *Candida albicans* as a fungus. Products **4a–l** (100 μ g) were dissolved in DMSO (1 mL), and 25 μ L of them was loaded to the paper disks (6 mm). Suspension of the microorganisms (100 μ L of 109 cell/ml) was diffused over sterile Muller Hilton Agar plates, and the paper disks were placed on the surface of culture plates. The minimum inhibitory concentration (MIC) of the products was determined by microdilution method and compared to the three commercial available antibiotics including chloramphenicol, gentamicin and nystatin. Unfortunately, none of compounds have antibiotic and antifungal activities among all tested bacteria and fungi.

Conclusion

In summary, SBA-Pr-SO₃H has employed as an efficient catalyst for the one-pot three-component synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. Some advantages of this method are environmentally friendly conditions, short reaction times, excellent yields and simple work-up procedures without using any chromatographic method for purification of products. Additionally, according to the computational and UV–Vis studies, it was found that some of the products are good candidates as UV absorbers due to their colorless crystal as well as broad and strong absorption in the range of UVB and UVA.

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