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A green procedure for one-pot synthesis of N,N′-alkylidene bisamides under solvent- and catalyst-free conditions

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Abstract. An effective solvent- and catalyst-free method is introduced for the synthesis of symmetrical N,N'-alkylidene bisamides as compounds with high usage in organic chemistry. In order to show the generality, the synthesis was done with various benzamides and aldehydes to obtain various derivatives in high yields.

1. Introduction

N,N'-alkylidene bisamides and their analogues have various applications in organic reactions and have attracted excessive attention of organic researchers. Some of these compounds are used as important biological and medicinal scaffolds [1,2]. Symmetrical and unsymmetrical N,N'-alkylidene bisamides are two major classes of these compounds with different synthetic routes. Some recent studies have reported the synthesis of bismides using Bronsted acids as catalyst [3-5]. Also, trifluromethanesulfonic acid has been used as a catalyst with high efficiency in preparation of symmetrical bisamides [6]. Zhu et al. reported the synthesis of fluorine-containing N,N'-alkylidene bisamides with fluoroalkane sulfonic acid [7]. Some other new methods for the synthesis of symmetrical N,N'-alkylidene bisamides and activated imines and aminal derivatives as potential precursors for the synthesis of β-amino acid in the presence of sulfamic acid, using Dean-Stark water trap, have also been reported by Selvam et al. and Milenkovic et al. [8,9]. In addition, Volochnyuk et al. and Bhatnagar et al. reported two other different methods; one through condensation of amides with carbonyl compounds in the presence of DMF-chlorotrimethylsilane as a catalyst system [10] and the other through syntheses of benzylidene bisamides by direct condensation of benzaldehyde and different amide derivatives [11].

Nowadays, synthetic methodologies based on green chemistry principles have attracted great attentions in organic synthesis. Using formic acid in Strecker and Mannich reactions [12,13]; using boric acid as a catalyst in the synthesis of a number of synthetic transformations, such as aza Michael addition to water [14], Biginelli reaction [15], thia Michael addition to water [16], esterification of ethyl acetoacetate, and decarboxylation of cyclic β-enaminoketoesters [17]; and condensation of imidazoles, amides, and aldehydes for getting arylated iminobenzamides with acid chlorides to give N-[alpha-(benzylidenamino)benzyl] benzamides and N,N'-benzylidenebis(benzamides) [18] are among the important methodologies for preparation of N,N'-alkylidene bisamides derivatives. Also, some other

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researchers reported bisamides synthesis by various catalysts [19]. In spite of all the researches to promote the synthesis procedures of N,N'-alkylidene bisamides, some disadvantages like expensive catalysts, low yield, difficult work up process, and intensive acidic condition are some limitations to the preparation of these compounds. Among the above-mentioned limitations, use of toxic and volatile solvents are the main problems, especially in terms of green chemistry principles. In this research, a solvent- and catalyst-free method is introduced for preparation of these compounds without any by-product and with easy work up procedure.

2. Experimental

2.1. Materials and methods

All solvents, reagents, and chemicals were purchased from Merck and Aldrich. Reactions and products were characterized by standard techniques. IR spectra were taken by a Shimadzu IR-470 spectrometer and melting points were taken by an Electrothermal 9100 apparatus. NMR spectra were recorded on an FT-NMR Bruker Ultra ShieldTM (500 and 300 MHz). An Agilent Technologies 5973 Mass Selective Detector (MS model) was used for EI-MS data.

2.2. General procedure for the synthesis of N,N′-alkylidenebisamides

In a dry test tube, a mixture of 1 mmol of aldehyde (benzaldehyde) and 2 mmol of amide (benzamide) was heated with stirring in an oil bath at 100°C under solvent-free condition. The progress of the reaction was followed by TLC analysis (eluent: n-hexane/ethylacetate, 3:2). After completion of the reaction, the product was settled down and recrystallized in ethanol (20 mL).

2.3. General procedure for the synthesis of 1, 1′(arylmethylen)-diurea

In a dry test tube, a mixture of 1 mmol of aldehyde (benzaldehyde) and 2 mmol of urea was heated with stirring in an oil bath at 100°C under solvent-free condition. The progress of the reaction was followed by TLC analysis (eluent: n-hexane/ethylacetate, 3:2). After completion of the reaction, the product was settled down and recrystallized in ethanol (20 mL).

2.4. Spectral data of representative compounds

N,N′-(phenylmethylene)dibenzamide (3a)

White solid, m.p: 235-237°C, IR (KBr): $\nu_{\max} = 3264$ (N-H), 3095 (C-H Ar), 2950 (C-H), 1652 (C=O amide), 1605 (C=C Ar), 1463 (C-N), cm^{-1}. MS (E. I.) (70 eV): m/z 209.

N,N′-(3-nitrophenylmethylene)dibenzamide (3i)

White solid, m.p: 233-235°C, IR (KBr): $\nu_{max} = 3311$, 3281 (N-H), 3085 (C-H Ar), 2977 (C-H aliphatic), 1649 (C=O amide), 1602 (C=C Ar), 1533, 1340 (NO_2), 1463 (C-N cm^{-1}. ^{1}H NMR (500 MHz, DMSO-d_6, TMS): δ (ppm): 9.18-9.19 (d, 2H), 8.29 (d, 1H), 8.15-8.17 (dd, 4H), 7.88-7.92 (m, 5H, Ar), 7.65-7.68 (t, 1H, Ar), 7.52-7.55 (m, 2H, Ar), 7.44-7.47 (m, 4H, Ar), 7.03-7.05 (t, 1H, Ar).

N,N′-(4-methoxyphenylmethylene) dibenzamide (3n)

Yellowish solid, m.p: 233-235°C, IR (KBr): $\nu_{\max} =$ 3272 (N-H), 3012 (C-H Ar), 2999 (C-H aliphatic), 1650 (C=O), 1579-1600 (C=C Ar), 1483 (C-N) cm.^{-1} MS (E. I.) (70 eV): m/z 237.

N,N′-(4-methylphenylmethylene) diurea (3q)

White solid, m.p: 240°C, IR (KBr): $\nu_{\max} = 3311$, 3440 (N-H, NH_2), 3028 (C-H Ar), 2977 (C-H aliphatic), 1652 (C=O amide), 1606 (C=C) cm^{-1}. ^{1}HNMR (500 MHz, DMSO-d_6, TMS): δ (ppm): 7.11-7.21 (q, 4H, Ar), 5.57-5.70 (d, 2H), 6.06-6.11 (t, 1H), 5.68 (s br, 3H), 5.45 (s br, 1H).

N,N′-(3-chlorophenylmethylene) diurea (3r)

White solid, m.p: 217-218°C, IR (KBr): $\nu_{\max} = 3233-$ 3415 (br, NH), 3051 (C-H Ar), 2979 (C-H aliphatic), 1650 (C=O, br), 1610 (C=C Ar), 1473 (C-N) cm^{-1}. ^{1}HNMR (500 MHz, DMSO-d_6, TMS): δ (ppm): 7.43-7.49 (m, 1H, Ar), 7.035-7.37 (d,1H, Ar), 7.23-7.30 (m, 2H, Ar), 6.71-6.77 (m, br, 2H), 6.28-6.34 (m, br, 1H), 5.52 (s. br, 1H), 5.35 (s, br, 1H).

N,N′-(4-CF$_3$-phenylmethylene) diurea (3s)

White solid, m.p: 240°C, ^{1}H NMR (300.1 MHz, DMSO-d_6, TMS): δ (ppm): 7.68-7.70 (d, 2H, Ar), 7.51-7.73 (d, 2H, Ar), 6.67-7.15 (d, br, 2H), 6.16-6.22 (s, br, 1H), 5.75 (s, br, 4H). ^{13}C NMR (75 MHz, DMSO-d_6). 58.8, 122.6, 125, 126.2, 126.8, 127.4, 127.8, 147.3, 147.6, 156, 157.7.

N,N′-(4-methoxyphenylmethylene) diurea (3v)

White solid, m.p: 193-195°C, ^{1}HNMR (500 MHz, DMSO-d_6, TMS): δ(ppm): 7.19-7.21 (d, 2H, Ar), 6.85-6.87 (d, 2H), 6.77 (s, br, 1H), 6.63-6.65 (d,1H), 6.07-6.10 (t, 1H), 5.58 (s, 2H), 3.71 (s, 3H, OCH_3).

3. Results and discussion

The reaction of benzaldehyde and benzamide was considered as the reaction model to optimize the reaction conditions (Scheme 1). Affording the desired product is shown in Figure 1.

In the first step, to investigate the effect of temperature on the synthesis of symmetrical N,N'-alkylidenebisamides, various temperatures were tested and the results are summarized in Table 1.

The results in Table 1 show that the best yields were obtained between 100 and 130°C; 100°C was selected as optimum temperature for this reaction.

Table 1. Optimization of temperature in the synthesis of symmetrical N,N'- alkylidenebisamides[a].

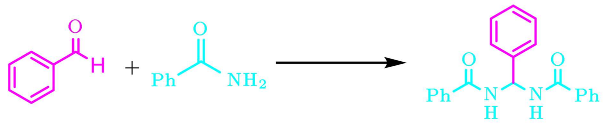
Entry	**Temperature (°C)**	**Time (h)**	**Yield (%)**
1	20	2	–
2	40	2	10
3	60	2	35
4	80	2	60
5	100	5 (min)	85
6	110	5 (min)	87
7	120	5 (min)	87
8	130	5 (min)	87

[a]benzaldehyde (1 mmol), benzamide (2 mmol), solvent-free.

Table 2. Effect of different solvents on the synthesis of symmetrical N,N'-alkylidenebisamides[a].

Entry	**Solvent**	**Amount (mL)**	**Time (h)**	**Yield (%)**
1	Ethyl acetate	1	6	45
2	CH3CN	1	6	60
3	H2O	1	6	-
4	PEG	1	6	-
5	Toluene	1	6	30
6	Solvent-free	-	5 (min)	85

[a]benzaldehyde (1 mmol), benzamide (2 mmol) catalyst-free



1a 2a 3a

Scheme 1. Reaction of benzamide (2 mmol) and benzaldehyde (1 mmol).

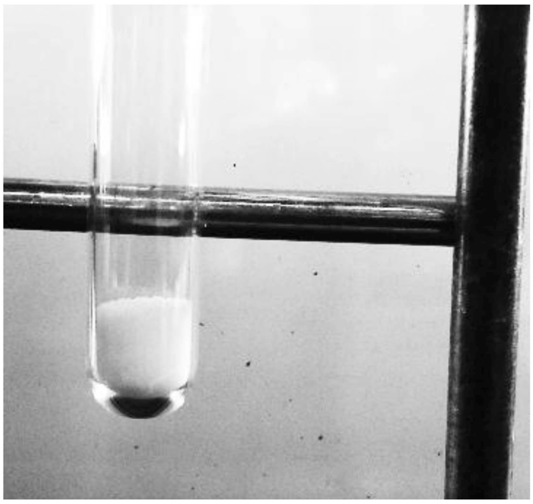


Figure 1. After completion of the reaction.

In the second stage, to determine the best solvent for the reaction, usual solvents were tested and the results are summarized in Table 2.

According to Table 2, there was not any product in the presence of H_2O and poly ethylene glycol as solvent. The best yield was obtained under solvent-free condition. After determining the best reaction conditions, the method was expanded to other derivatives and the results are listed in Scheme 2 and Table 3.

The results show that both benzamide and acetamide react with aromatic aldehydes in short reaction time with high yield. In the first step of the mechanism, nucleophilic addition of amide to aromatic aldehyde produces an imine as an intermediate and then, with another nucleophilic addition of amide to carbon of imine, the final product is formed (Scheme 3). It is noteworthy that the scope of the reaction was carried out with aliphatic aldehydes, but desirable product was produced in low yield.

Solvent-free condition is an advantage of this method, which shows its efficiency in comparison with other reported procedures for the synthesis of these compounds. Table 4 compares the presented method with other reported methods in the literature [22,25,29,30-32]. In terms of green chemistry, the use of catalysts and toxic solvents in some of the above procedures is considered as undesirable problem, which is resolved in our method.

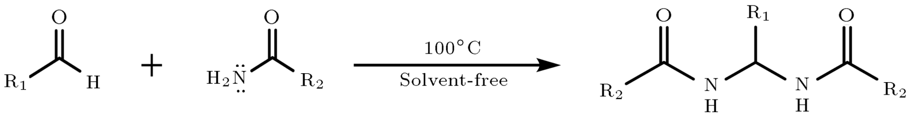
4. Conclusion

An effective solvent- and catalyst-free approach was introduced for the synthesis of symmetrical N,N'-alkylidenebisamides with good yields. The products were characterized by melting points, FT-IR, ^{1}H NMR, and ^{13}C NMR. Considering the green chemistry principles, removal of the catalysts and solvent from the reaction, which are usually toxic and hazardous reagents, is the main advantage of this method in

Table 3. Synthesis of *N*,*N*′-alkylidene bisamide derivatives under catalyst- and solvent-free conditions[a].

Entry	R1	R2	Product	Time (min)	Yield (%)	MP (°C)	MP (°C) [Ref]
1	(1a)	Ph	(2a)	5	85	235-237	235-38 [20]
2	(1b)	Ph	(2b)	10	80	245-247	246 [21]
3	(1c)	Ph	(2c)	10	83	251-253	252-254 [20]
4	(1d)	Ph	(2d)	10	85	243-245	240-243 [22]
5	(1e)	Ph	(2e)	10	88	249-250	252-255 [22]
6	(1f)	Ph	(2f)	10	75	240-242	240-242 [20]
7	(1g)	Ph	(2g)	10	80	256-259	257-261 [22]
8	(1h)	Ph	(2h)	15	85	238-240	243-245 [20]
9	(1i)	Ph	(2i)	15	80	233-235	232-235 [20]
10	(1j)	Ph	(2j)	10	82	268-270	266-268 [23]
11	(1k)	Ph	(2k)	30	75	201-203	201-202 [24]
12	(1l)	Ph	(2l)	30	85	233-235	230-232 [25]
13	(1m)	H	(2m)	20	70	234-236	235-238 [20]
14	(1b)	CH3	(2n)	20	78	264-267	265-267 [20]
15	(1j)	CH3	(2o)	20	75	243-245	244-246 [20]
16	(1a)	NH2	(2p)	5	85	201-203	205-07 [26]
17	(1b)	NH2	(2q)	20	88	210-213	210-213 [26]
18	(1p)	NH2	(2r)	10	80	217-218	188-190 [26]
19	(1q)	NH2	(2s)	5	90	198-201	198-201 [26]
20	(1g)	NH2	(2t)	5	83	193-195	195-197 [27]
21	(1i)	NH2	(2u)	20	85	203-205	200 [28]
22	(1l)	NH2	(2v)	15	80	193-195	193-194 [27]
23	(1n)	NH2	(2w)	10	74	188-190	182-183 [29]
24	(1n)	Ph	N.R(2x)	—	—	—	—
25	(1o)	Ph	N.R(2y)	—	—	—	—

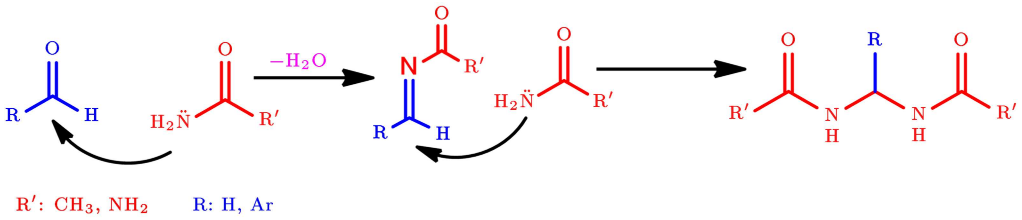
[a] aldehyde (1 mmol), amide (2 mmol), 100 (°C)



1a-o R₂: Ph, CH₃, NH₂ 2a-y

R₁: a = Ph b = 4-MeC₆H₅ c = 4-BrC₆H₅ d = 4-CNC₆H₅
e = 4-FC₆H₅ f = 2-ClC₆H₅ g = 4-ClC₆H₅ h = 2,4-diClC₆H₄
i = 3-NO₂C₆H₅ j = 4-NO₂C₆H₅ k = 3-MeOC₆H₅
l = 4 MeOC₆H₅ m = HCOH n = 4-OHC₆H₅
o = 4-Me₂NC₆H₅ p = 3-ClC₆H₅ q = CF₃C₆H₅

Scheme 2. List of alkylidenebisamides derivatives.



Scheme 3. Suggested mechanism for the synthesis of symmetrical *N*,*N*′- alkylidenebisamides.

Table 4. Comparison of diverse methods for the synthesis of symmetrical N,N'-alkylidenebisamides.

Entry	Catalyst	Solvent	Temperature (°C)	Yield (%)	Ref.
1	SBSSANPs	Toluene	Reflux	93	[22]
2	Boric acid	Toluene	Reflux	92	[25]
3	p-TSA	-	100	91	[29]
4	MSA	Etylacetate	90	91	[30]
5	BF_3 $^*OEt_2$	Acetone	RT	82	[31]
6	Nano-$SnCl_4.SiO_2$	n-Hexane	Reflux	90	[32]
7	-	-	100	90	This work

comparison with other reported procedures. Simplicity of work up process, short reaction time, and high yield are other remarkable advantages of this method.

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