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N,*N*,*N*′,*N*′-Tetramethylethylene-diaminium-*N*,*N*′-disulfonic acid trifluoroacetate and pyridinium-*N*-sulfonic acid hydrogen sulfate as highly effective dual-functional catalysts for the preparation of *N*,*N*′-alkylidene bisamides

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Abstract: In this research, Brønsted-acidic ionic liquids *N*,*N*,*N*′,*N*′-tetramethylethylene-diaminium-*N*,*N*′-disulfonic acid trifluoroacetate ([TMEDSA][$TFA]_2$) and pyridinium-*N*-sulfonic acid hydrogen sulfate ([Py-SO_3H][HSO_4]) have been introduced as dual-functional catalysts for the green, simple and effective preparation of *N*,*N*′-alkylidene bisamides by the reaction of primary amides (2 eq.) with arylaldehydes (1 eq.) under solvent-free conditions. The reaction results and conditions of the catalysts have been compared with the previously reported ones. [TMEDSA][$TFA]_2$ and [Py-SO_3H][HSO_4] were superior to the previously reported catalysts in terms of two or more of these factors: reaction times (10–45 min), yields (86–98%), temperature and the reaction conditions. Additionally, a plausible and attractive mechanism based on dual functionality of the catalysts has been proposed.

Keywords: Brønsted-acidic ionic liquid; *N*,*N*,*N*′,*N*′-tetramethylethylene-diaminium-*N*,*N*′-disulfonic acid trifluoroacetate ([TMEDSA][$TFA]_2$); *N*,*N*′-alkylidene bisamide; pyridinium-*N*-sulfonic acid hydrogen sulfate ([Py-SO_3H][HSO_4]); solvent-free.

1 Introduction

Ionic liquids (ILs) have attracted great attention for their use as attractive reaction media, reagents and catalysts in organic synthesis. The inimitable properties of ILs, which caused their wide applications in this field, include thermal and chemical stability, non-volatility, non-flammability, broad temperature range of the liquid state, aptitude to solve a wide range of compound classes, appropriate electrochemical window, catalyzation of various organic transformations and their capability to be functionalized for different purposes [1–10]. Among the ILs, Brønsted acids have been particularly found to be effective catalysts in several organic syntheses [4–10].

In many organic transformations, utilization of toxic and volatile organic solvents is inevitable, which is not satisfactory from a green chemistry point of view. An effectual way to solve this problem is to perform reactions under solvent-free conditions. This can also enhance yield and selectivity, saves energy and time, increases reaction rates, makes synthesis easier and prevents or minimizes waste and by-products [11–15].

Amide and bisamide functionalities are part of the framework of many pharmacological, biological, chemical and industrial molecules [16–23]. Bisamides are vital moieties for introducing *gem*-diaminoalkyl fragments in retro-inverse pseudo-peptide derivatives [19, 20]. Additionally, these compounds are used in the synthesis of peptidomimetic materials [21, 22], and as ligands in the Ullmann reaction leading to potentially pharmacologically active compounds [23]. Bisamides are also applied as antitumor [24] and insecticide [25] agents. Applications in polymerizations [26] and in the separation of xylene isomers [27] have also been reported. A literature survey shows that a practical method for the preparation of *N*,*N*′-alkylidene bisamides includes the reaction of primary amide (2 eq.) with aldehydes (1 eq.). Various catalysts have been reported for this synthesis, such as hydroxyapatite [28], phosphotungstic acid [29], nano-$SnCl_4{\cdot}SiO_2$ [30], $H_{14}[NaP_5W_{29}MoO_{110}]$ [31], $B(HSO_4)_3$ [32], $NiFe_2O_4$@SiO_2-PPA [33], CH_3COCl [34], HPVAC-20 (heteropoly-11-tungsto-1-vanadophosphoric acid supported on activated natural clay) [35], $ZnCl_2/SiO_2$ [36] and sulfonated carbon/nano-titania composite (C/TiO_2-SO_3H) [37]. Nevertheless, many of the reported methods have restrictions such as, for example, long reaction times, moderate yields, harsh reaction conditions and the utilization of volatile and harmful organic solvents.

In this research, we have used two Brønsted-acidic ILs, namely *N*,*N*,*N*′,*N*′-tetramethylethylene-diaminium-*N*,*N*′-disulfonic acid trifluoroacetate ([TMEDSA][$TFA]_2$)

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and pyridinium-*N*-sulfonic acid hydrogen sulfate ([Py-SO_3H][HSO_4]), as highly efficient and general dual-functional catalysts for the solvent-free preparation of *N*,*N*′-alkylidene bisamides from primary amides (2 eq.) and arylaldehydes (1 eq.), thereby overcoming several of the above-mentioned limitations.

2 Results and discussion

To find optimal reaction conditions, the condensation of benzamide (2 mmol) and benzaldehyde (1 mmol) was chosen as a model reaction (Scheme 1) and investigated in the presence of different amounts of [TMEDSA][$TFA]_2$ or [Py-SO_3H][HSO_4] in the temperature range of 80–100°C under solvent-free conditions. These results are summarized in Table 1. As shown in Table 1, the reaction was efficiently performed using 10 mol.% of [TMEDSA][$TFA]_2$ or 15 mol.% of [Py-SO_3H][HSO_4] at $T = 90$°C (Table 1, entries 3 and 8). Increasing the amounts of catalyst or the temperature slightly reduces the reaction times in the case of both catalysts (Table 1, entries 4, 6, 9 and 11), and the yield slightly enhanced in the case of [Py-SO_3H][HSO_4] (Table 1, entries 9 and 11). Nevertheless, 90°C was selected as the optimal temperature for both catalysts. Moreover, 10 and 15 mol.% were chosen as the optimal catalyst amounts for [TMEDSA][$TFA]_2$ and [Py-SO_3H][HSO_4], respectively. These conditions were deliberately chosen because performing reactions using less catalyst at lower temperatures is more in compliance with green-chemistry protocols. The solvent-free reaction was also examined at $T = 90$°C in the absence of a catalyst whereby the reaction did not significantly progress even after long reaction time (Table 1, entry 1).

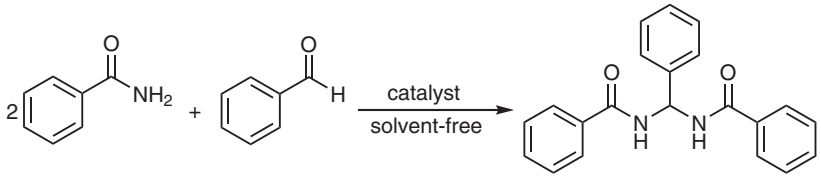
After finding the optimal reaction conditions, amides were reacted with various aldehydes using [TMEDSA][$TFA]_2$ or [Py-SO_3H][HSO_4] as catalysts in order to appraise their scope and effectiveness. The respective results are depicted in Table 2. As is shown there, benzamide, acetamide and acrylamide in the presence of both catalysts afford the products in high yields and short reaction times. Furthermore, [TMEDSA][$TFA]_2$ and [Py-SO_3H][HSO_4] effectively catalyze the reaction when benzaldehyde and arylaldehydes possess electron-withdrawing, electron-releasing substituents or halogens in *ortho*, *meta* or *para* positions. Nevertheless, [TMEDSA][$TFA]_2$ gave the desired *N*,*N*′-alkylidene bisamides in often higher yields and in shorter reaction times as compared to [Py-SO_3H][HSO_4]. According to the results reported in Table 2, both ILs are highly efficient and general catalysts for the synthesis.

Table 1: Effect of the catalyst quantity and temperature on the model reaction shown in Scheme 1.

Entry	Catalyst	Amount of catalyst (mol.%)	Temperature (°C)	Time (min)	Yield (%)[a]
1	–	–	90	100	Trace
2	[TMEDSA][$TFA]_2$	5	90	30	85
3	[TMEDSA][$TFA]_2$	10	90	20	96
4	[TMEDSA][$TFA]_2$	13	90	16	96
5	[TMEDSA][$TFA]_2$	10	80	25	82
6	[TMEDSA][$TFA]_2$	10	100	15	96
7	[Py-SO_3H][HSO_4]	13	90	60	87
8	[Py-SO_3H][HSO_4]	15	90	40	93
9	[Py-SO_3H][HSO_4]	20	90	35	94
10	[Py-SO_3H][HSO_4]	15	80	65	76
11	[Py-SO_3H][HSO_4]	15	100	32	94

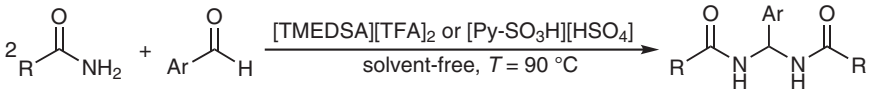
[a]Isolated yields.

The ILs are dual-functional catalysts having both acidic (SO_3H) and basic sites (trifluoroacetate and hydrogen sulfate are weak bases; see Figs. 1 and 2) [5, 9]. Thereby [TMEDSA][$TFA]_2$ and [Py-SO_3H][HSO_4] can be used as efficient, general catalysts for reactions which need both acidic and basic catalysts simultaneously, for example, the synthesis of *N*,*N*′-alkylidene bisamides. This topic is elaborated in a proposed reaction mechanism with [TMEDSA][$TFA]_2$ as catalyst (Scheme 2). First, amide is activated by the basic group of [TMEDSA][$TFA]_2$ and added to the activated aldehyde by the acidic site of the catalyst to afford intermediate **I** (trifluoroacetate assists to bind a proton from the amide NH_2). Then, the acidic IL helps to remove a molecule of H_2O from **I** to give intermediate **II** (trifluoroacetate assists for removing



Scheme 1: The model reaction used for the screening of the reaction conditions.

Table 2: The synthesis of *N*,*N*′-alkylidene bisamides catalyzed by $[TMEDSA][TFA]_2$ (10 mol.%) or $[Py\text{-}SO_3H][HSO_4]$ (15 mol.%).



Comp. no.	R	Ar	[TMEDSA][TFA]$_2$		[Py-SO$_3$H][HSO$_4$]		M.p. (°C) (Lit.)
			Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	
1a	C_6H_5	C_6H_5	20	96	40	93	216–218 (217–219 [31])
1b	C_6H_5	4-$O_2NC_6H_4$	13	96	30	97	260–262 (259–261 [32])
1c	C_6H_5	3-$O_2NC_6H_4$	10	98	40	93	230–232 (232–235 [32])
1d	C_6H_5	2-$O_2NC_6H_4$	25	92	35	90	258–260 (256–257 [32])
1e	C_6H_5	4-ClC_6H_4	20	98	40	95	250–252 (252–254 [37])
1f	C_6H_5	2,4-$Cl_2C_6H_3$	30	91	30	87	196–198 (199–200 [37])
1g	C_6H_5	3-BrC_6H_4	15	92	30	90	224–226 (224–226 [33])
1h	C_6H_5	4-FC_6H_4	10	95	20	95	227–229 (226–228 [29])
1i	C_6H_5	4-$CH_3OC_6H_4$	35	93	40	92	228–230 (230–232 [29])
1j	C_6H_5	4-$CH_3C_6H_4$	25	95	35	89	239–241 (242–244 [33])
1k	CH_3	C_6H_5	25	90	35	88	235–237 (238–240 [34])
1l	CH_3	4-$O_2NC_6H_4$	20	94	35	91	272–274 (270–272 [33])
1m	CH_3	4-$CH_3C_6H_4$	35	88	40	90	271–273 (270–272 [34])
1n	CH_2=CH	4-$O_2NC_6H_4$	40	91	45	86	240–242

[a]Isolated yields.

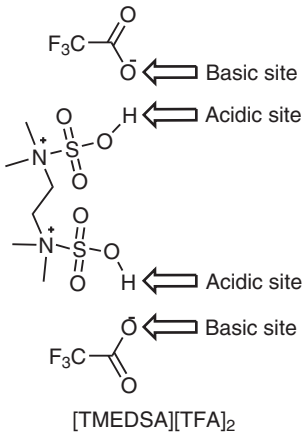


Fig. 1: The acidic and basic sites of $[TMEDSA][TFA]_2$.

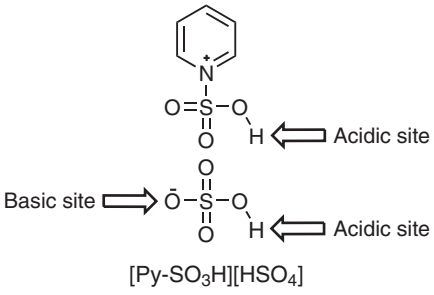
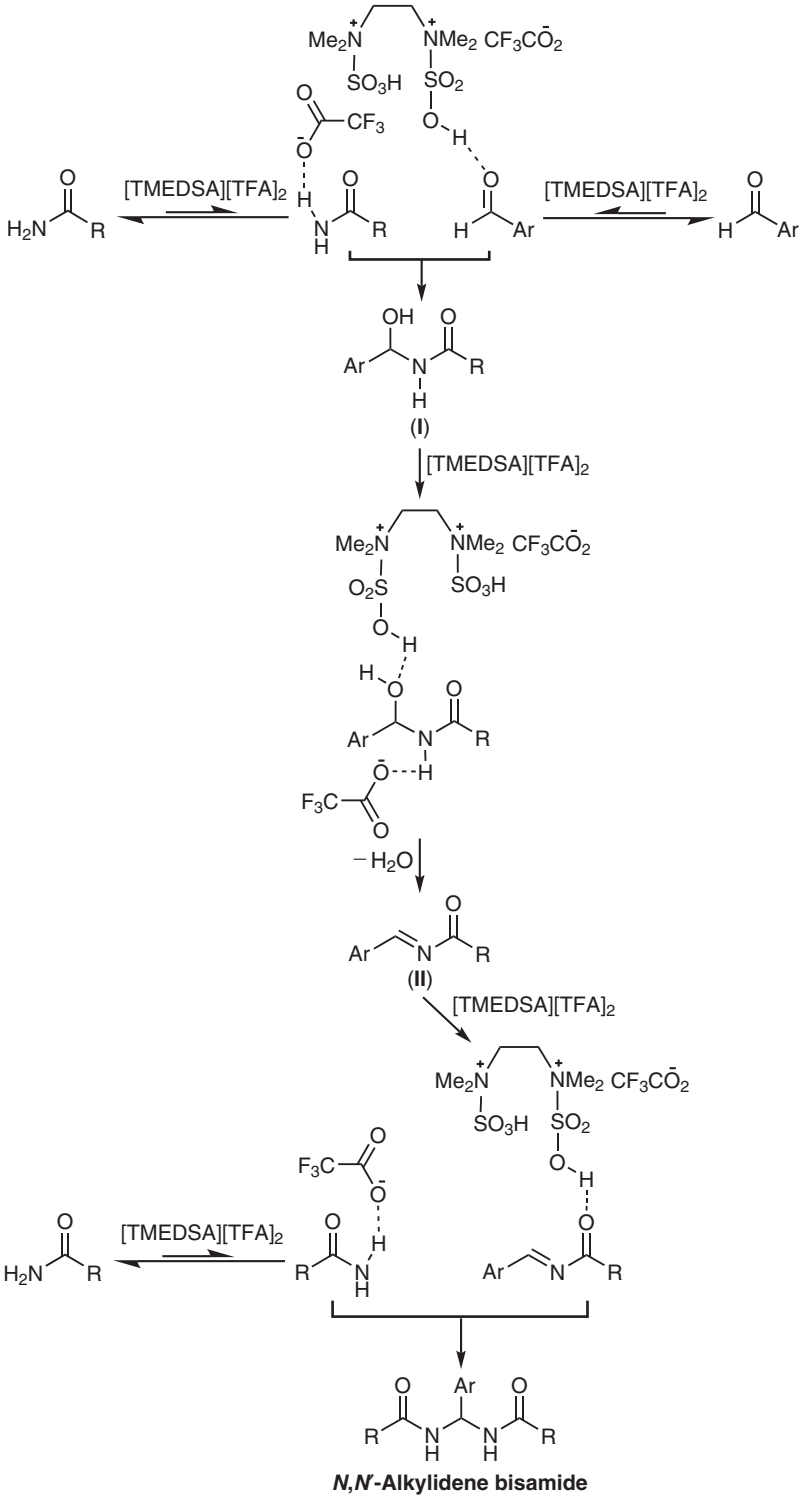


Fig. 2: The acidic and basic sites of $[Py\text{-}SO_3H][HSO_4]$.

H_2O by attracting a proton). Intermediate **II** is activated by the catalyst to accept a nucleophile, whereupon the second amide (which is activated by the catalyst anion) is added



Scheme 2: The proposed mechanism for the synthesis of *N*,*N*′-alkylidene bisamides.

to it leading to the product. [Py-SO_3H][HSO_4] can also catalyze the reaction in this way. The mechanism is supported by the literature [33]. In fact, high efficacy of both catalysts can be attributed to dual functionality of them, and capability to activate the electrophiles and the nucleophiles to accelerate all steps of the reaction mechanism. Furthermore, the higher effectiveness of [TMEDSA][$TFA]_2$ with respect to [Py-SO_3H][HSO_4] can be related to having more basic sites (two instead of one, respectively) while both ILs have two acidic sites (see Figs. 1 and 2).

Table 3: Results and reaction conditions obtained with $[TMEDSA][TFA]_2$ and $[Py\text{-}SO_3H][HSO_4]$ in comparison with previously reported catalysts.

Catalyst	Conditions	Time range	Yields (%)	Ref.
$[TMBSED][TFA]_2$	Solvent-free, 90°C	10–40 min	88–98	–
$[Py\text{-}SO_3H][HSO_4]$	Solvent-free, 90°C	20–45 min	86–97	–
Hydroxyapatite	CH_3CN, reflux	3 h	87–95	[28]
Phosphotungstic acid	Toluene, reflux	20–72 h	44–94	[29]
Nano-$SnCl_4{\cdot}SiO_2$	*n*-Hexane, reflux	0.81–5.1 h	71–96	[30]
$H_{14}[NaP_5W_{29}MoO_{110}]$	CH_3OH, reflux	50–120 min	42–95	[31]
$B(HSO_4)_3$	Solvent-free, 100°C	15–45 s	83–96	[32]
$NiFe_2O_4$@SiO_2-PPA	CH_3OH, reflux	40–130 min	52–93	[33]
CH_3COCl	CH_3CN, r.t.	5–25 min	80–98	[34]
HPVAC-20	Solvent-free, 110°C	25–45 min	85–96	[35]
$ZnCl_2/SiO_2$	Solvent-free, 80°C	25–40 min	70–89	[36]
$C/TiO_2\text{-}SO_3H$	Solvent-free, 100°C	0.5–3 h	80–94	[37]

Finally, we compared the effectiveness and the reaction conditions of our catalysts with those reported in the literature, the results being shown in Table 3. The data clearly demonstrate that $[TMEDSA][TFA]_2$ and $[Py\text{-}SO_3H][HSO_4]$ are superior to the reported catalysts in terms of two or more of these factors: reaction times, yields, temperature and/or reaction conditions.

3 Conclusion

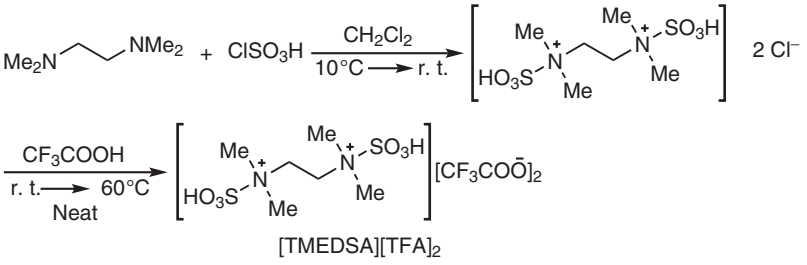
In summary, we have reported the efficient synthesis of *N*,*N*′-alkylidene bisamides in the presence of two Brønsted-acidic ILs, namely *N*,*N*,*N*′,*N*′-tetramethylethylenediaminium-*N*,*N*′-disulfonic acid trifluoroacetate and pyridinium-*N*-sulfonic acid hydrogen sulfate. The advantages of the presented methodologies over previous ones are effectiveness, generality, high yields, relatively short reaction times, cleaner reaction profile, utilization of solvent-free conditions, ease of product isolation and purification, simple preparation and dual-functionality of the catalysts and good compliance with green-chemistry protocols.

Although in our previous work we were able to regenerate similar catalysts as employed here [38], we believe that the preparation of them from fresh *N*,*N*,*N*′,*N*′-tetramethylethylene-diamine or pyridine is more economic, greener and easier than their regeneration.

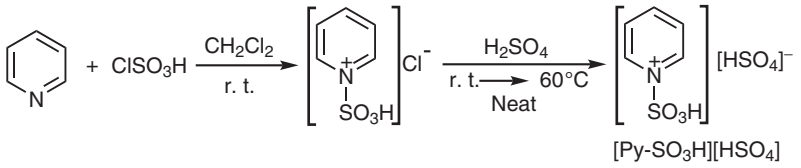
4 Experimental section

4.1 General

All chemicals were purchased from Merck or Fluka Chemical Companies. $[TMEDSA][TFA]_2$ and $[Py\text{-}SO_3H][HSO_4]$ were synthesized according to reported procedures (Schemes 3 and 4) [9, 10]. All known compounds were identified by comparison of their melting points and/or spectral data with those reported in the literature. Melting points were recorded on Büchi B-545 apparatus in open capillary tubes. Progress of the reactions was monitored by thin-layer chromatography (TLC) using silica gel SIL G/UV 254 plates. FT-IR spectra were run using Shimadzu IR-60 apparatus. ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) were run on Bruker Avance DPX, FT-NMR spectrometers (δ in ppm). Mass spectra were recorded on a 5975C VL MSD model with its triple-axis detector.



Scheme 3: The preparation of $[TMEDSA][TFA]_2$.



Scheme 4: The synthesis of [Py-SO_3H][HSO_4].

4.2 General procedure for the preparation of *N,N′*-alkylidene bisamides

A mixture of amide (2 mmol), arylaldehyde (1 mmol) and [TMEDSA][$TFA]_2$ (0.050 g, 0.10 mmol) or [Py-SO_3H][HSO_4] (0.038 g, 0.15 mmol) was vigorously stirred by a small rod at $T = 90$°C. After the reaction was completed as monitored by TLC, the reaction mixture was cooled to room temperature, and the resulting precipitate was recrystallized from EtOH (96%) to give the pure product.

4.3 Selected spectral data of the synthesized *N,N′*-alkylidene bisamides

4.3.1 *N,N′*-(4-Chlorophenylmethylene)dibenzamide (1e)

^{1}H NMR (500 MHz, DMSO-d_6, 25°C, TMS): $\delta = 7.03$ (t, $J = 7.6$ Hz, 1H, methine CH), 7.45–7.58 (m, 7H, H_{Ar}), 7.57 (t, $J = 7.3$ Hz, 2H, H_{Ar}), 7.94 (d, $J = 7.3$ Hz, 4H, H_{Ar}), 9.10 (d, $J = 7.4$ Hz, 2Hm NH). –^{13}C NMR (125 MHz, DMSO-d_6, 25°C, TMS): $\delta = 58.9$, 128.2, 128.8, 128.9, 129.2, 132.3, 132.9, 134.3, 139.9, 166.3.

4.3.2 *N,N′*-(4-Fluorophenylmethylene)dibenzamide (1h)

^{1}H NMR (500 MHz, DMSO-d_6, 25°C, TMS): $\delta = 7.04$ (t, $J = 7.6$ Hz, 1H, methine CH), 7.22 (t, $J = 8.8$ Hz, 2H, H_{Ar}), 7.49 (t, $J = 7.8$ Hz, 4H, H_{Ar}), 7.53–7.58 (m, 4H, H_{Ar}), 7.94 (d, $J = 7.4$ Hz, 4H, H_{Ar}) 9.09 (d, $J = 7.6$ Hz, 2H, NH). –^{13}C NMR (125 MHz, DMSO-d_6, 25°C, TMS): δ (ppm) 58.9, 128.2, 128.9, 129.2, 129.3, 132.2, 134.4, 137.1, 137.2, 161.3, 163.3, 166.3.

4.3.3 *N,N′*-(4-Methylphenylmethylene)dibenzamide (1j)

^{1}H NMR (500 MHz, DMSO-d_6, 25°C, TMS): $\delta = 2.30$ (s, 3H, CH_3), 7.02 (t, $J = 7.7$ Hz, 1H, methine CH), 7.20 (d, $J = 8.0$ Hz, 2H, H_{Ar}), 7.37 (d, $J = 8.1$ Hz, 2H, H_{Ar}), 7.49 (t, $J = 7.3$ Hz, 4H, H_{Ar}), 7.56 (t, $J = 7.3$ Hz, 2H, H_{Ar}), 7.92 (d, $J = 7.8$ Hz, 4H, H_{Ar}), 8.99 (2H, d, $J = 7.8$ Hz, NH). –^{13}C NMR (125 MHz, DMSO-d_6, 25°C, TMS): $\delta = 21.3$, 59.2, 127.0, 128.1, 128.9, 129.4, 132.2, 134.5, 137.5, 138.0, 166.1.

4.3.4 *N,N′*-(4-Nitrophenylmethylene)diacrylamide (1n)

IR (KBr): $\nu = 1221$, 1346, 1505, 1557, 1625, 1665, 3270 cm^{-1}. –^{1}H NMR (500 MHz, DMSO-d_6, 25°C, TMS): $\delta = 5.69$ (dd, $J = 1.7$, 10.4 Hz, 2H, vinyl CH), 6.19 (dd, $J = 1.7$, 17.2 Hz, 2H, vinyl CH), 6.36 (d, $J = 10.2$ Hz, 1H, vinyl CH), 6.39 (d, $J = 10.2$ Hz, 1H, vinyl CH), 6.77 (t, $J = 7.6$ Hz, 1H, methine CH), 7.64 (d, $J = 8.7$ Hz, 2H, H_{Ar}), 8.27 (d, $J = 8.7$ Hz, 2H, H_{Ar}), 9.07 (d, $J = 7.6$ Hz, 2H, NH). –^{13}C NMR (125 MHz, DMSO-d_6, 25°C, TMS): $\delta = 57.8$, 124.0, 127.1, 128.3, 131.4, 147.5, 147.7, 164.6. –MS (EI, 70 eV): m/z (%) = 275 (0.4) $[M]^+$.

5 Supplementary information

Selected original spectra of the synthesized *N,N′*-alkylidene bisamides have been given in the Supplementary information available online (https://doi.org/10.1515/znb-2019-0064).

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