Indian Journal of Chemistry
Vol. 53B, November 2014, pp 1436-1441

Synthesis of some novel class of bisisoxazolidine derivatives *via* 1,3-dipolar cycloaddition reactions in water

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Received 20 December 2013; accepted (revised) 16 April 2014

Simultaneous double 1,3-dipolar cycloaddition reactions of glyoxal derived bisnitrones have been described in water. Significant rate acceleration and improved yields of exclusively diastereoselective and regioselective novel bisisoxazolidines in water have been observed at room temperature in a short reaction time compared with conventional solvents.

Keywords: Bisnitrones, double cycloaddition reaction, novel bisisoxazolidines, diastereoselectivity, regioselectivity, water

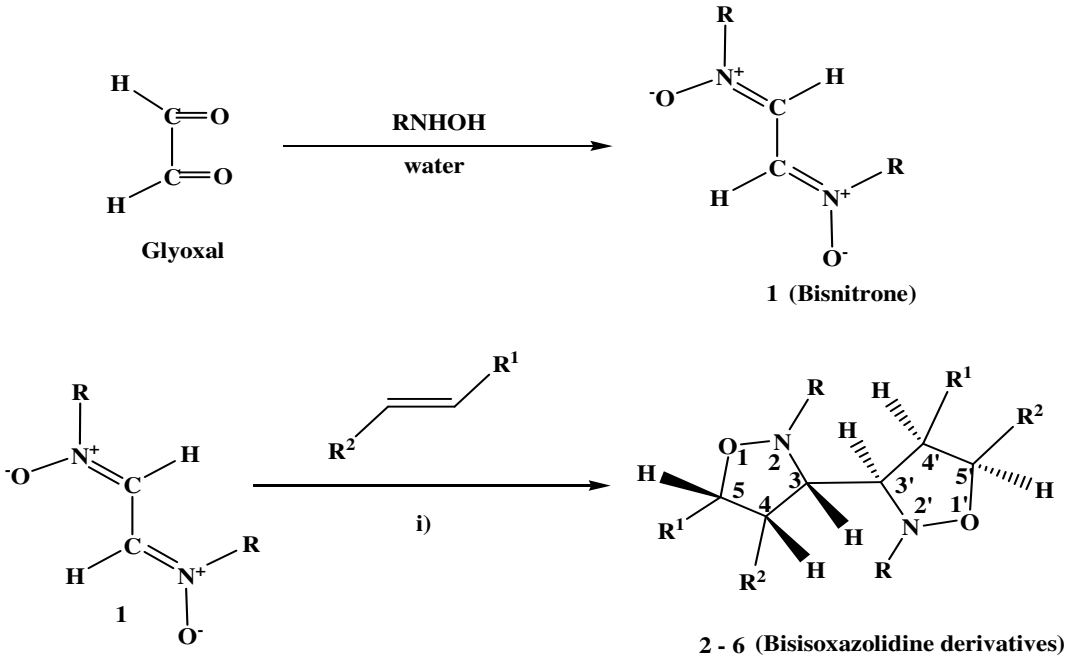
1,3-Dipolar cycloaddition reactions are an integral and weighty part of organic chemistry in pedagogy and research as well. Cycloaddition methodology used in aqueous media has brought forth a number of heterocyclic compounds, usually with a regio and stereoselectivity pecularity. These heterocycles include isoxazoles, isoxazolidines and pyrrolidines. The rate of acceleration of organic reactions in aqueous media has been considered as due to one or a combination of the following factors and phenomena[1-3], the high cohesive energy density of water, the high internal pressure within the medium, the hydrogen-bonding ability, the hydrophobic packing of diene and dienophile in cycloaddition reactions, the hydrophobic vs. antihydrophobic effects, the micellar catalysis, the solvophobicity, and the solvent polarity. The concept of ''on-water'' reactions[4,5] was introduced by Sharpless in **2005**. The ''on water'' method consists simply of stirring the reactant(s) with water to generate an aqueous suspension and it has been observed that both kinetics and yields are extremely enhanced in most cases, compared with those in organic solvents.

In continuation of our efforts to establish green methodologies in nitrone cycloaddition reactions[6-9], we report herein a new route to the synthesis and 1,3-dipolar cycloaddition reactions of glyoxal derived bisnitrones (having vast synthetic potentials) with a variety of alkenes to produce novel bisisoxazolidine derivatives **(2-6)** in water **(Scheme I)**. This is quite a new approach of synthesis of nitrone from glyoxal. The present study has been carried out with three different maleimides (N-methyl/phenyl/benzyl) and ethyl acrylate, styrene respectively in water. Simultaneously the reactions have been also studied in organic solvent (CH_2Cl_2).

Results and Discussion

Dipolarophiles have been classified into water-super and water-normal on the basis of the magnitude of their rate of response towards water. A ketone (C=O) conjugated to an alkene or alkyne is a water-super dipolarophile. Esters, ethers and aryl rings conjugated to an alkene are water-normal dipolarophiles. Almost all the reactions in water are very fast (3-4 hr in case of maleimides, methyl acrylate and 5 hr for styrene) compared to the normal cycloaddition reactions in organic solvents which were reported to take longer periods (26-48 hr)[10,11].

It is possible that water promotes the reaction through hydrogen bond formation with the carbonyl oxygen atom of the α,β-unsaturated carbonyl compounds and thereby increasing the eletrophilic character at the β-carbon which is attacked by nucleophilic oxygen atom of the nitrone. Thus water activates maleimide, methyl acrylate and thereby greatly facilitates the reaction. Reaction rate is comparatively slower in styrene because of very lesser possibility of the formation of hydrogen bonding between water and alkenes but still the rate of the reaction and the yield is higher than the cycloaddition reactions performed in solvents like THF, CH_2Cl_2 **(Table I)**. An explanation have been suggested for these results in terms of the frontier



i) water, RT, 3-5 hr, N_2 atmosphere

R = CH_3 ; C_6H_5 ; $CH_2C_6H_5$

2 : R^1, R^2 = -CONMeCO-

3 : R^1, R^2 = -CONPhCO-

4 : R^1, R^2 = -CONCyCO-

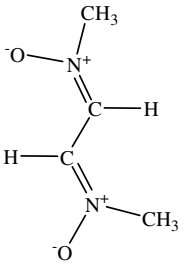
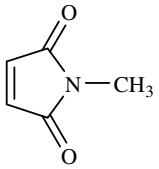
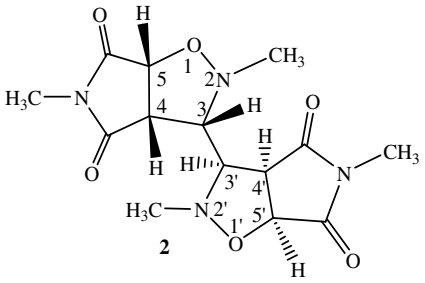
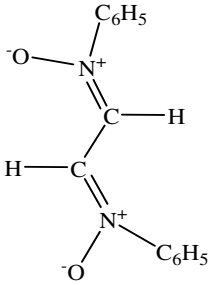
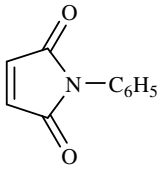
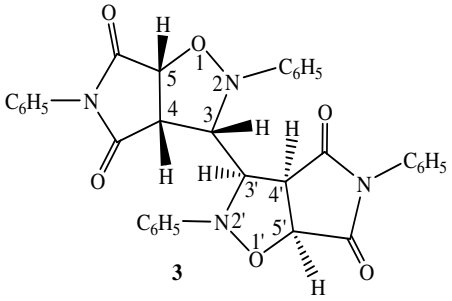
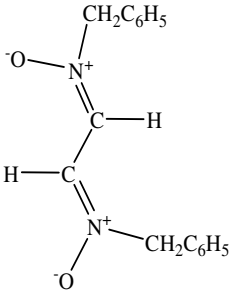
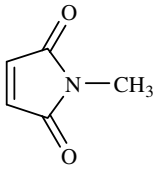
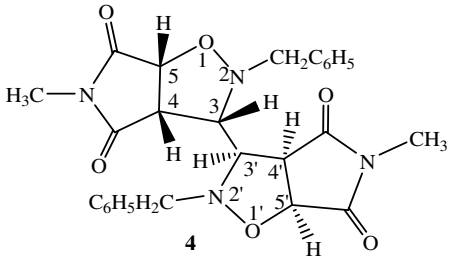
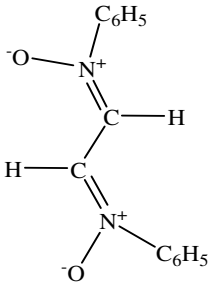
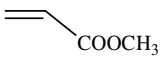
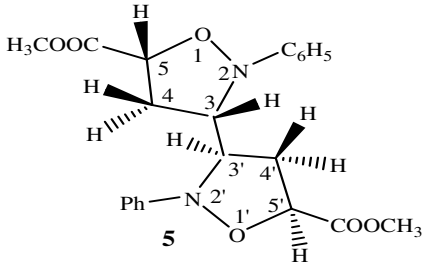
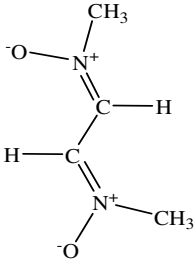
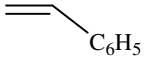
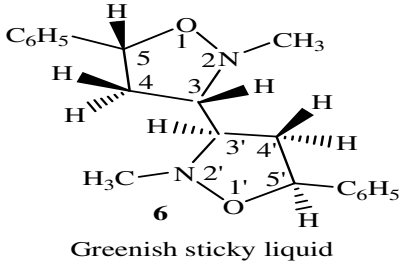
5 : R^1 = H ; R^2 = $-CO_2CH_3$

6 : R^1 = H ; R^2 = $-C_6H_5$

Scheme I — Synthesis of bisnitrone and bisisoxazolidine derivatives from glyoxal

molecular orbital (FMO) theory which has been used extensively to explain regioselectivity and to predict the yield, rate in 1,3-dipolar cycloadditions[12,13]. This theory states that the Gibbs energy of activation is related to the energy gap between the interacting HOMO and LUMO. The dipolarophiles like styrene is weak hydrogen bond acceptor, which means that their FMO's are only slightly affected by hydrogen bond interactions and lead to a reduction of the energy gap between the interacting FMO's (in this case, the HOMO of the dipolarophile and LUMO of the 1,3 dipole). Consequently, the Gibbs energy of activation of the reaction is reduced and the reaction is accelerated in water with good yield. Bisnitrones **1** reacted with *N*-substituted maleimides giving bisisoxazolidines. Diastereoselective reactions of the dipole **1** furnished diastereoselective cycloadducts **2-4** and are classified as *trans trans* biscycloadducts as the 3-H and 4-H protons on each isoxazolidine ring are *trans* orientated as evidenced from ^{1}H NMR spectroscopy[14,15]. On the other hand, bisnitrones **1** reacted with methyl acrylate and styrene giving exclusively regioselective bisisoxazolidines **5-6**. All the novel biscycloadducts **2-6** are obtained as diastereoselective and regioselective isomeric forms and stereochemical informations portrayed in the drawing implies relative and not absolute relations[16]. The structures of the diastereoselective and regioselective (5-substituted) novel bisisoxazolidine derivatives are confirmed on the basis of ^{1}H NMR spectroscopy[14,15]. It is also evident from the ^{1}H NMR spectrum of the diastereoselective bisisoxazolidines **2-4** that the structures are expected to be symmetrical in nature and that 3-H, 4-H are *cis* orientated on both rings whilst vicinal coupling constant has been found to be $J_{3,4}$ ~ 6.80 Hz[17]. Compared to conventional conditions, the cycloaddition reactions performed in water are much faster and selective[18]. As an example, the reaction between nitrone **1** and alkenes, afforded bisisoxazolidine **2** at RT after 26 hr in 62% yield in

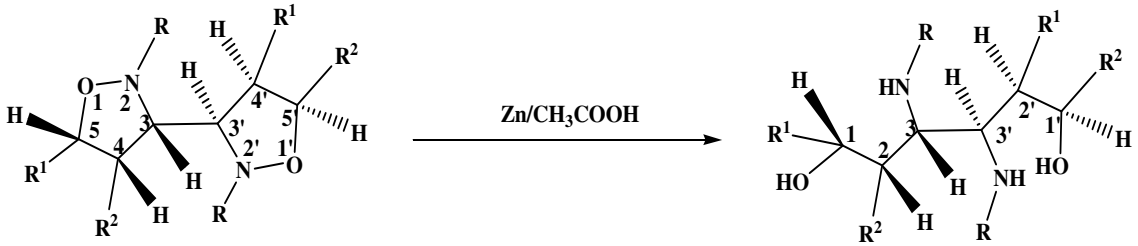
Table I — 1,3-dipolar cycloaddition reaction of glyoxal derived bisnitrones with alkenes in water

Entry	Bisnitrone[a] (**1**)	Alkene	Bisisoxazolidine[b] (**2-6**)	Time (hr)	Yield[c] (%)
1			 **2** de = 88 ; Yellowish white crystals, m.p 142 ^{0}C	3 (26)	94 (62)
2			 **3** de = 96, Yellow solid, m.p 116^0 C	3 (27)	91 (59)
3			 **4** de = 92 ; White crystals, m.p 135^0 C	4 (28)	91 (60)
4			 **5** Colourless liquid	4 (30)	88 (60)
5			 **6** Greenish sticky liquid	5 (35)	83 (57)

[a]Reaction conditions: bisnitrone (1 mmol), alkenes (2 equivalent), water (15 mL), N_2 atmosphere

[b]All products were characterized by IR, ^{1}H NMR, ^{13}C NMR and MS spectral data.

[c]Isolated yield after purification. Figures in parentheses indicate reactions performed in conventional solvents (CH_2Cl_2).



2 (Bisisoxazolidine derivatives) **2a (Bis 1,3-amino alcohols)**

Scheme II — Synthesis of bis 1,3-amino alcohols

CH_2Cl_2 and after 3 hr in 94% yield in water **(entry 1)** respectively. The reaction of nitrone **1** with various alkenes follow the general mechanistic pattern of 1,3-dipolar cycloaddition reactions as found in literature[10,11]. Initial study reports on the biological activity of the synthesized bisisoxazolidine derivatives are also very encouraging. All the novel bisisoxazolidine derivatives **2-6** have been found to be very effective against both gram positive and gram negative organisms which give an opportunity to develop new broad spectrum antimicrobial agents.

Furthermore, these novel biscycloadducts **2-6** are found to have vast synthetic potential as they could be converted into 1,3 difunctional amino alcohols **(Scheme II)**. Studies are in progress.

All the biscycloadducts are found to be stable and have prominent molecular ion peak and base peaks in the mass spectrum as expected. It has been observed that the *N*-methyl dipole reacts less selectively but furnishes higher yields than its electron poor *N*-phenyl analogue. A plausible stereochemistry of the bisisoxazolidines obtained from maleimides **2-4** has been assigned on the basis of 3-H and 4-H proton signals of both isoxazolidine rings appeared as double doublet and doublets respectively[19,20].

In addition, these bisisoxazolidine derivatives could be easily recovered on work-up. Since the products are fairly soluble in water, they could be easily extracted with ether. Important signals of C_3H, C_4H and C_5H protons of both the isoxazolidine rings (*cis*, *cis*) of the novel bisisoxazolidine derivatives have been found to be merged and obtained as a single signal. Double doublet signal of C_4H protons appeared as broad signal in majority of the novel biscycloadducts and coupling constant values could not be calculated. High selectivity is observed in these simultaneous double cycloaddition reactions and best selectivity (diastereomeric excess) was observed in the cycloaddition reactions of *N*-phenylbisnitrone with *N*-phenyl maleimide (*de*% 96, **entry 2, Table I**). Enhanced reaction rates, excellent yields, and high selectivity are the features observed in these double cycloaddition reactions. All the products were characterized by ^{1}H NMR, ^{13}C NMR, IR and MS spectroscopic data.

Experimental Section

^{1}H NMR spectra were recorded with a Bruker DRX 300 spectrometer (300 MHz, FT NMR) using TMS as internal standard. ^{13}C NMR spectra were recorded on the same instrument at 75 MHz. The coupling constants (*J*) are given in Hz. IR spectra were obtained with a Perkin-Elmer RX 1-881 machine as film or as KBr pellets for all the products. MS spectra were recorded with a Jeol SX-102 (FAB) instrument. All the reactions were monitored by TLC using 0.25 mm silica gel plates (Merck $60F_{254}$ UV indicator) while column chromatography was performed with silica gel (E.Merck India) 60–200 mesh. All other reagents and solvents were purified after receiving from commercial suppliers. *N*-Benzylhydroxylamine, *N*-Phenyl maleimide, *N*-Methyl maleimide, starting materials, reagents used in the reactions were obtained commercially from Aldrich, Lancaster, Fluka and were used without purification, unless otherwise indicated. Characterization of the novel biscycloadducts have been confirmed on the basis of spectral data.

General procedure for the synthesis of nitrone 1

To a solution of glyoxal (309 mg, 5.3127 mmole) in diethyl ether (20 mL) *N*-methylhydroxylamine (500 mg, 2 equivalent) and anhydrous $MgSO_4$ (2 g) was added. The reaction mixture was kept at RT with constant stirring with a magnetic stirrer under N_2 atmosphere for 8 hr. The formation of bisnitrone was monitored by TLC (R_f = 0.36). The reaction mixture was filtered and the filtrate on concentrated *in vacuo*

furnished *N*-methyl bisnitrone as white crystals (86%; m.p: 78°C). Same methodology was followed for the synthesis of other bisnitrones (R = C_6H_5; $CH_2C_6H_5$). All the bisnitrones were found stable and were reacted with various activated alkenes in 1,3-dipolar cycloaddition reactions in water at room temperature.

Spectroscopic data for nitrone **1** (R = CH_3): UV: λ_{max} 233 nm. IR (K Br): 1635 (m), 1610 (s) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 6.45 (d, 1H, *J* = 3.22 Hz, -CH=N^+), 6.23 (d, *J* = 3.22 Hz,-CH=N^+), 3.84 (s, 6H, 2 × CH_3, N^+-CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.60 (CH=N^+), 140.94 (CH=N^+), 24.74, 24.70 (N^+-CH_3).

General procedure of synthesis of diastereoselective bisisoxazolidine derivatives in water (Table I; entry 1)

N-methylmaleimide (2 equivalent) was added to a solution of bisnitrone (1 equivalent ; R = CH_3) in water (15 mL) and the reaction mixture was stirred at RT for an appropriate time **(Table I)**. After completion of reaction, as indicated by TLC (R_f = 0.68, 0.62), the reaction mixture was extracted with diethyl ether (3 × 10mL), the organic layer was washed with saturated brine (2 × 15mL), dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The resulting crude products were directly charged on silica gel column and eluted with a mixture of ethyl acetate:n-hexane (1:6) to afford pure bisisoxazolidines **2** (**Table I, entry 1**, 94% and 6% respectively) as yellowish white crystals. Same methodology was followed for other substrates depicted in **Table I**.

Both the major and minor bis diastereomers gave satisfactory ^1H NMR, ^{13}C NMR, MS, IR and elemental analyses data. Spectral data of the major bis diastereomers are represented as follows.

Spectral data of diastereomeric bisisoxazolidine derivatives (2-4)

(3R, 3aR, 6aS)-dihydro-3-((3′S, 3′aS, 6aR)-hexahydro-2,5-dimethyl-4, 6-dioxo-2*H*-pyrrolo-[3,4-d]isoxazol-3-yl)-2′, 5′-dimethyl-2*H*-pyrrolo-[3,4-d]isoxazole-4,6(5*H*, 6a*H*) dione 2

2: Yellowish white crystals, Yield 94%; R_f = 0.68; FT-IR (K Br): 2820 (m), 1760 (s), 1675 (s), 1465 (m), 1230 (m), 1125 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 3.31 (d, 2H, *J* = 4.06 Hz, 2 × C_5H), 3.10 (s, 6H, 2 × $ONCH_3$), 2.99 (s, 6H, 2 × (O=C)NCH_3), 2.85 (d, 2H, *J* = 4.22 Hz, 2 × C_3H), 2.50 (dd, br, 2H, 2 × C_4H); ^{13}C NMR ($CDCl_3$): δ 174.78, 173.12 (carbonyl carbons), 75.80 (C_5, $C_{5'}$), 69.94 (C_3, $C_{3'}$), 56.77 (C_4, $C_{4'}$), 26.63, 26.58 (methyl carbons); FAB-MS:*m/z* 338 (M^+), 169, 168, 154. Calcd. for $C_{14}H_{18}O_6N_4$: C, 49.68; H, 5.36; N, 16.56. Found: C, 49.53; H, 5.25; N, 16.44%.

(3R, 3aR, 6aS)-dihydro-3-((3'S, 3′aS, 6aR)-hexahydro-4,6-dioxo-2,5-diphenyl-2*H*-pyrrolo[3,4-d]isoxazol-3-yl)-2′, 5′-diphenyl-2*H*-pyrrolo[3,4-d]isoxazole-4,6(5*H*, 6a*H*)dione, 3

3: White crystals, Yield 91%; R_f = 0.66; FT-IR (K Br): 3025 (m), 2830 (m), 1764 (s), 1660 (s), 1485 (m), 1345 (m), 784 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.36-7.26 (m, 10H, 2 × (O=C)NC_6H_5), 6.62-6.50 (m, 10H, 2 × ONC_6H_5), 2.11 (dd, br, 2H, 2×C_4H), 1.85 (d, 2H, *J* = 6.00 Hz, 2 × C_5H), 1.67 (d, 2H, *J* = 6.10 Hz, 2 × C_3H); ^{13}C NMR ($CDCl_3$): δ 172.40, 172.26 (carbonyl carbons), 138.83, 138.12, 137.94, 137.71, 129.74, 129.70, 129.33, 129.04 (aromatic carbons), 76.15 (C_5, $C_{5'}$), 66.47 (C_3, $C_{3'}$), 55.80 (C_4, $C_{4'}$); FAB-MS: *m/z* 586 (M^+), 293, 292, 216, 77. Calcd. for $C_{34}H_{26}O_6N_4$: C, 69.60; H, 4.46; N, 9.55. Found: C, 69.50; H, 4.38; N, 9.49%.

(3R, 3aR, 6aS)-2-benzyl-3-((3′S, 3′aS, 6aR)-2′-benzyl-hexahydro-5-methyl-4,6-dioxo-2*H*-pyrrolo-[3,4-d]isoxazol-3-yl)-dihydro-5-methyl-2*H*-pyrrolo-[3,4-d]isoxazole-4,6(5*H*, 6a*H*)dione4. **4**: White crystals, Yield 91%; R_f = 0.62; FT-IR (KBr): 3010 (m), 2900 (m), 1760 (s), 1660 (s), 1482 (m), 1340 (m), 780 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.46-7.26 (m, 10H, 2 × $CH_2C_6H_5$), 4.37 (d, 2H, *J* = 7.16 Hz, 2 × C_5H), 3.24 (d, 2H, *J* = 7.14 Hz, 2 × C_3H), 2.89 (dd, br, 2H, 2 × C_4H), 2.60 (s, 6H, 2 × N-CH_3 protons), 2.15 (s, 4H, 2 × $CH_2C_6H_5$); ^{13}C NMR ($CDCl_3$): δ 177.18, 177.04 (carbonyl carbons), 133.22, 133.12, 132.90, 132.70 (aromatic carbons), 73.67 (C_5, $C_{5'}$), 64.80 (C_3, $C_{3'}$), 53.77 (C_4, $C_{4'}$), 32.05, 31.94 (benzyl carbons), 28.70, 28.58 (N-Me carbons); FAB-MS: *m/z* 490 (M^+), 245, 244, 154, 77. Calcd. for $C_{26}H_{26}O_6N_4$: C, 63.64; H, 5.34; N, 11.42. Found: C, 63.57; H, 5.26; N, 11.35%.

General procedure of synthesis of regioselective bisisoxazolidine derivatives in water (Table I; entry 4)

Methyl acrylate (2 equivalent) was added to a solution of bisnitrone (1 equivalent ; R = C_6H_5) in water (15 mL) and the reaction mixture was stirred at RT for an appropriate time (**Table I**). After completion of reaction, as indicated by TLC (R_f = 0.76), the reaction mixture was extracted with diethyl ether (3 × 10 mL), the organic layer was washed with saturated brine (2 × 15 mL), dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The resulting crude product was directly charged on silica gel column and eluted

with a mixture of ethyl acetate:n-hexane (1:6) to afford pure bisisoxazolidine **5** (**Table I, entry 4**, 88%) as colourless liquid. Same methodology was followed for other substrate depicted in **Table I.**

Spectral data of regioselective bisisoxazolidine derivatives (5-6)

(3S,5S)-methyl-3-(((5′R)-5-(methoxycarbonyl)-2-phenylisoxazolidine-3-yl)methyl)-2'-phenyl isoxazolidine-5′-carboxylate 5

5: Colorless gummy liquid, Yield 88%; R_f = 0.58; FT-IR (K Br): 3026 (m), 2890 (m), 1760 (s), 1664 (s), 1485 (m), 783 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 8.07-8.02 (m, 5H, C_6H_5), 7.52-7.45 (m, 5H, C_6H_5), 3.72 (dd, 2 × 1H, *J* = 5.44, 5.40 Hz, C_4H, *endo*), 3.43 (s, 2×3H, $-COOCH_3$), 2.96 (d, 2H, *J* = 6.32 Hz, 2 × C_5H), 2.59 (d, 2H, *J* = 6.30 Hz, 2 × C_3H), 1.24 (ddd, 2 × 1H, *J* = 2.80, 2.82 Hz, C_4H); ^{13}C NMR ($CDCl_3$): δ 170.24, 170.15 (carbonyl carbons), 129.47, 129.38, 129.25, 129.17 (aromatic carbons), 70.46 (C_5, $C_{5'}$), 60.54 (C_3, $C_{3'}$), 52.49 (C_4, $C_{4'}$), 17.22, 17.07 (ester methyl carbons); FAB-MS: *m/z* 412 (M^+), 206, 205, 147, 129, 77, 59. Calcd. for $C_{22}H_{24}O_6N_2$: C, 64.05; H, 5.86; N, 6.79. Found: C, 63.97; H, 5.74; N, 6.70%.

(3S,5S)-2-methyl-3-(((5′R)-2′-methyl-5-phenylisoxazolidin-3-yl)methyl)-5′-phenylisoxazolidine 6

6: Greenish thick liquid, Yield 83%; R_f = 0.52; FT-IR (K Br): 3215 (m), 2905 (m), 2245 (s), 1484 (m), 780 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.88-7.73 (m, 5H, C_6H_5), 7.50-7.44 (m, 5H, C_6H_5), 3.60 (ddd, 2 × 1H, *J* = 6.24, 6.22 Hz, C_4H, *endo*), 2.76 (d, 2H, *J* = 6.06 Hz, 2 × C_5H), 2.62 (d, 2H, *J* = 6.28 Hz, 2 × C_3H), 2.30 (s, 2 × 3H, N-Me protons), 1.70 (dd, 2 × 1H, *J* = 3.66, 3.62 Hz, C_4H); ^{13}C NMR ($CDCl_3$): δ 136.67, 136.58, 136.52, 136.38, 131.80, 131.72, 131.55, 131.23 (aromatic carbons), 73.60 (C_5, $C_{5'}$), 58.45 (C_3, $C_{3'}$), 55.37 (C_4, $C_{4'}$), 36.64, 35.21 (N-Me carbons); FAB-MS: *m/z* 324 (M^+), 246, 161, 147, 77; Calcd. for $C_{20}H_{24}O_2N_2$: C, 74.03; H, 7.45; N, 8.64. Found: C, 73.95; H, 7.33; N, 8.59%.

Conclusion

In conclusion, a new route to the synthesis of bisisoxazolidines from glyoxal derived bisnitrones via one pot double cycloaddition reactions with maleimides and other activated alkenes have been reported. *N*-methyl dipoles are found to be more reactive, but less selective than their *N*-phenyl analogues. Biscycloadducts with *N*-methyl/*N*-phenyl substituents on the isoxazolidine rings are found to be *cis* disposed with respect to 3-H and 4-H protons. Finally, we have also shown that these cycloaddition reactions may be conveniently carried out in water for the synthesis of corresponding novel bisisoxazolidines in good conversions and yields with high synthetic potentials, selectivities.

Acknowledgements

The authors are pleased to acknowledge the financial support from the Department of Science & Technology, Government of India, New Delhi (grant no:SR/S1/OC-34/2011). The authors are equally grateful to SAIF-CDRI, Lucknow for providing spectral data.

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