Short communication

Synthesis of novel D-glucose based anionic bolaamphiphiles and their catalytic application in 1,3-dipolar nitrone cycloaddition reactions

Vikash Kumar [a], Amrita Chatterjee [a], Biswajit Gopal Roy [b], Mainak Banerjee [a,*]

[a] Department of Chemistry, BITS Pilani, K. K. Birla Goa Campus, Goa 403 726, India
[b] Department of Chemistry, School of Physical Sciences, Sikkim University, 6th Mile, Tadong, Gangtok, Sikkim 737 102, India

ABSTRACT

Three D-glucose based bolaamphiphiles have been synthesized by a simple and efficient synthetic methodology utilizing the free C-3 hydroxyl group of diisopropylidene glucose. The resulting bolaamphiphiles showed unique aggregation behavior in aqueous solution, e.g. transformation of vesicle to tubular morphology on aging. The chiral hydrophobic interior of these vesicles was utilized as tiny reaction vessels to carry out 1,3-dipolar nitrone cycloadditions in water. The corresponding isoxazolidine derivatives were formed with high diastereoselectivity in several cases. The bolaamphiphile with 12 carbon spacer (**5c**) showed better diastereoselectivity among the rest with just 2 mol% of catalyst loading and can be reused.

© 2017 Elsevier B.V. All rights reserved.

1. Introduction

In recent years, bolaamphiphiles [1–3] have progressively gained importance because of their abilities to provide original supramolecular structures [4] and advanced biomaterials [5]. A large variety of bolaamphiphiles (cationic, anionic, nonionic, zwitterionic) of different structures have been reported, so far [6–13]. Out of them, sugar-based bolaamphiphiles [9–13] are of special interest for pharmaceutical and other applications because of their natural origin, biocompatibility and biodegradability. A large variety of non-ionic [9,10] and cationic [11–13] sugar-based bolaamphiphiles have been reported so far with wide variety of properties and applications, e.g. formulation of anti-HIV combination therapy [9], anticancer drug delivery [10], gene transfection [11], bacterial agglutination [12] etc. However, there are very few reports available on sugar-based anionic bolaamphiphiles and their applications [14]. Although bolaamphiphiles have been used for a wide range of applications, another potential application of these molecules that remains practically unexplored is their ability to catalyze reactions in aqueous media [15]. The bolaamphiphiles have all the properties of a normal surfactant and they are also capable of forming micelles or vesicles in water. The in-built chirality of sugar-based bolaamphiphiles could be advantageous in order to get improved stereoselectivity in various organic transformations. Being chiral these ionic bolaamphiphiles

are expected to show unique aggregate morphologies as well [11,13, 14].

Nitrones are important reaction intermediates and are useful for the synthesis of several therapeutic agents e.g. antifungal [16], antitubercular [17], cytotoxic [18], DNA intercalator [19] etc. These therapeutic agents commonly have isoxazolidine backbone. Nitrones are mostly used as the dipole in 1,3-dipolar cycloaddition reaction to form isolated or fused isoxazole or isoxazolidine nucleus [20,21], which is mostly dependent on alkyne or alkene counterparts as dipolarophile like alkyl acrylates. The cycloaddition reactions show high regio- and stereoselectivity; different mechanistic pathways have been proposed in favor of stereoselectivity [22,23]. Notably, most of the conventional methods to synthesize the pharmacologically important heterocyclic skeleton via nitrone cycloaddition use hazardous reagents, toxic solvents and harsh reaction conditions [24–26]. However, few green methods are also available that include the use of commercial surfactants as catalysts [27], ionic liquid [28], microwave condition [29], mechanochemical methods [30] etc. The present work is inspired by Chatterjee (one of the authors of the present work) and co-workers in which they described that use of sucrose as a useful additive to induce high diastereoselectivity to isoxazolidine derivatives in a model reaction [27]. We thought, the built-in chirality of the D-glucose based bolaamphiphiles would play a pivotal role in imparting higher regioselectivity and stereoselectivity to the final product mixture. In addition, lower CMC of bolaamphiphiles would ensure that the reaction works at much lower catalyst loading. In this endeavor, we describe the development of a synthetic route to a new series of D-glucose derived anionic

* Corresponding author.
E-mail addresses: kumarniper@gmail.com (V. Kumar), amrita@goa.bits-pilani.ac.in (A. Chatterjee), bgroy@cus.ac.in (B.G. Roy), mainak@goa.bits-pilani.ac.in (M. Banerjee).

bolaamphiphiles having sulphonate as the polar head group (**5a–c**). In addition, their surface properties and catalytic activity in 1,3-dipolar nitrone cycloaddition have been reported.

2. Results and discussion

D-Glucose was converted to the initial building blocks (**2a–c**) via formation of 1,2:5,6-di-*O*-isopropyledene glucofuranoside (**1**) in high yields adopting a procedure developed by us [31]. They were converted to unstable aldehydes (**3a–c**) by deprotection followed by cleavage of corresponding diols with NaIO$_4$. The aldehydes were converted to alcohols (**4a–c**) by in-situ reduction using NaBH$_4$ (Scheme 1). In the final step, the bolaamphiphiles were synthesized by treatment of alcohols (**4a–c**) with 1,3-propane sultone to produce the anionic bolaamphiphiles (**5a–c**) in good overall yields.
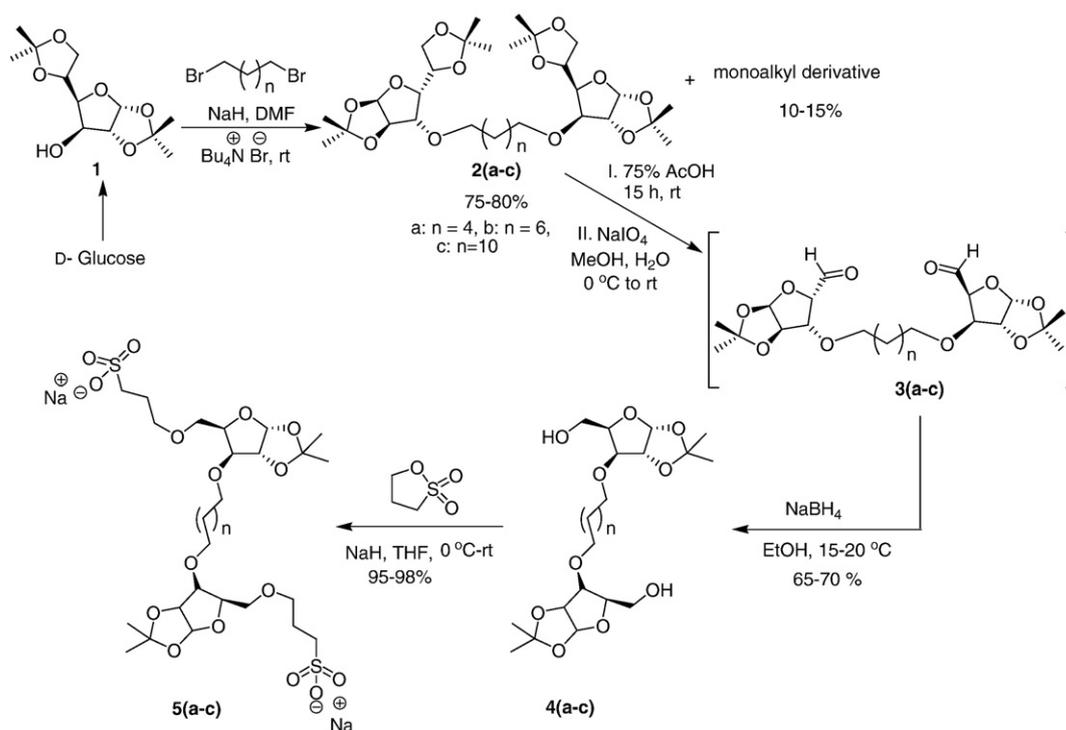
The structures of the new bolaamphiphiles (**5a–c**) were confirmed by ^{1}H NMR, ^{13}C NMR, ESI-MS and CHN analysis. The formation of bolaamphiphile alcohols (**4a–c**) were confirmed by appearance of a 2H doublet at ~4.5–4.6 and a mass peak at *m/z* 485 [M + 23]$^+$ for **4a**, at *m/z* 513 [M + 23]$^+$ for **4b** and at *m/z* 569 [M + 23]$^+$ for **4c**, respectively. Appearance of a two proton multiplet at δ 1.81–1.94 for **5a**, 1.78–1.97 for **5b** and 1.78–1.97 for **5c** due to —CH$_2$—SO$_3^-$ and a two proton triplet at δ 2.87 for **5a**, 2.84 for **5b** and 2.85 for **5c** due to –O—CH$_2$CH$_2$CH$_2$ SO$_3^-$ in ^{1}H NMR and appearance of a peak at δ 48.0 (—CH$_2$SO$_3^-$) for all **5a**, **5b** and **5c** in ^{13}C NMR and their corresponding ESI-MS peaks at 352, 366 and 394 (*m/z*, z = 2; −ve mode) for **5a**, **5b** and **5c**, respectively confirm the product formation.

At first, the CMC values of the new bolaamphiphiles (**5a–c**) were calculated from the conductivity versus concentration plots (for details see SI, Fig. S1). It is considered that the CMC values from conductometric measurements are obtained from the bulk solution which is unaffected by any interfacial events. The CMCs were further validated by steady-state fluorescence experiments using pyrene as the probe (for details see SI, Fig. S2). Bolaamphiphiles are generally having low CMC values

as compared to their corresponding conventional surfactants [32]. They indeed showed lower CMC values as expected. The observed CMC of surfactants **5a**, **5b** and **5c** from conductivity measurements are 0.0076 mM, 0.0054 mM and 0.0038 mM, respectively. The CMCs of **5a**, **5b**, and **5c**, obtained from steady-state fluorescence measurements were found as 0.0052 mM, 0.0041 mM, and 0.0028 mM, respectively, which closely matched with the CMCs obtained by conductometric measurements. A general trend was observed: the CMC decreases with increasing chain length [33]. The lower value of CMC for **5c** compared to others is a direct consequence of increased overall hydrophobicity due to longer spacer length. The observed CMC values were of the same order of magnitude of previously reported sugar based bolaamphiphiles [33].

The self-assembly of the bolaamphiphiles **5a–c** occurs rapidly under neutral condition at a concentration of 1 mg per mL of water. Room temperature sonication was sufficient to ensure the formation of stable supra-molecular assembly in aqueous solution. TEM analysis [34] revealed that vesicles formation occurs in case of all bolaamphiphiles (Fig. 1A, and Fig. S3(A–C) of SI). It was also found that initially they form vesicles that transform into tubular morphologies upon standing the aqueous solution of bolaamphiphiles for 7 days (Fig. 1B and Fig. S3(D,E) of SI). Furthermore, in some cases, twisted ribbon like morphology was also observed (Fig. S3F, SI) for **5c**. Similar morphology was observed for **5c** by SEM analysis as well (Fig. S4(A,B) of SI). The molecular aggregation of anionic bolaamphiphiles, to build up tubular and ribbon like morphology, is presumably driven by hydrophobic interactions between the alkyl chains.

To standardize the reaction condition for catalytic applications of bolaamphiphiles, the formation of the emulsion droplets were confirmed by taking optical micrograph of different bolaamphiphiles containing aqueous solutions of reactants before reaction would actually proceed (Fig. 2A). This ensures that the proposed reaction can be carried out in this organized media. Dynamic light scattering (DLS) experiment of the solution containing bolaamphiphile (**5c**), *m*-nitrobenzaldehyde and phenylhydroxylamine was also performed which confirmed that

Scheme 1. Synthetic route to D-glucose derived bolaamphiphiles **5(a–c)**.
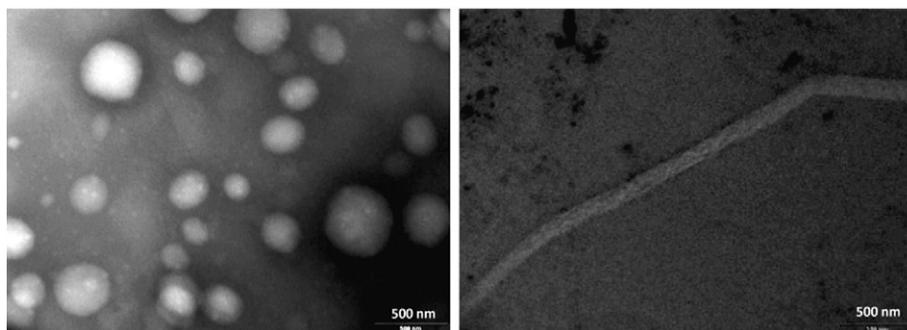
Fig. 1. TEM images of **5c**: (A) formation of vesicles after 1 day aging of the solution; (B) tubular morphology after 7 days aging of the solution.

the size of emulsion droplets is mostly in few hundreds of nanometer (Fig. 2B). These droplets act as confined reactor to carry out the organic transformation inside their core.

To establish a suitable condition for water exclusion reaction, we screened catalytic activity of all three bolaamphiphiles (**5a–c**) for model reactions between one electron deficient aldehyde (viz. *m*-nitrobenzaldehyde) and one electron rich aldehyde (viz. *p*-methoxybenzaldehyde) with phenylhydroxylamine and compared the results with the reported yields by Chatterjee et al. (Table 1) [27]. In this regard, the same model reactions were repeated by us using SDS as catalyst adopting the reported condition [27]. The complete condensation of phenylhydroxylamine and aromatic aldehydes on an average took 1 h to afford intermediate nitrones (**6**). Once the nitrone completely formed, ethyl acrylate was added in-situ to the reaction mixture to carry out the cycloaddition reaction and it was allowed to stir at room temperature till the reaction got completed (Table 1). As mentioned in table 1, yields are generally very good (75–90%). As expected, the reactions were successfully carried out with just 2 mol% of catalyst loading. It is noteworthy to mention that the amount of SDS used in the method reported by Chatterjee et al. was 5 fold [27]. This is because of low CMC of synthesized bolamaphiphiles which allows formation of adequate numbers of vesicles or emulsion droplets in solution even at low concentrations. In addition, significant increase in the reaction rate was observed for bolaamphiles as compared to SDS (Table 1). In general, the reactions produced 5-substituted products (**9**) preferably over 4-substituted products (**8**) for each of the bolaamphiphiles (**5a–c**). For 4-substituted products (**8**) the reaction is stereospecific in favor of endo isomer for all cases (Table 1, entries 1–3, 11–13). For 5-substituted products (**9**), unpredictable diastereoselectivity was observed with bolaamphiphile **5c**, whereas, other bolaamphiphiles do not impose much diastereoselectivity. However, to our dismay, the synthesized bolaamphiphiles occasionally failed to achieve the regioselectivity and

diastereoselectivity that are shown by SDS (Table 1, entries 6, 14). Based on these observations bolaamphiphile **5c** was chosen for further study with an expectation of better diastereoselective implications. In a separate study, the concentration of bolaamphiphile **5c** was changed from 2 mol% to 5 mol% and 10 mol% on a model reaction with *m*-nitrobenzaldehyde and found that increased catalyst concentration does not make any significant improvement in terms of reaction time, yield and stereoselectivity (Table 1, entries 4, 5).

After establishing the standard reaction condition, a series of aromatic aldehydes was treated with phenylhydroxylamine and then corresponding nitrone was treated with ethyl acrylate in the presence of 2 mol% of bolaamphiphile **5c** as catalyst in a single-pot. The methodology was found to be excellent in terms of yields and reaction time. Both aldehydes with electron withdrawing group (EWG) as well as electron donating groups (EDG) equally participated in the reaction to produce corresponding isoxazolidines in high yields (80–90%). As expected, reactions involving aromatic aldehydes with EWG completed at a faster rate (Table 2, entries 1–3), whereas, aromatic aldehydes with EDG took longer time for complete conversion. The structures of the isoxazolidine derivatives (**8** and **9**) were established by ^{1}H NMR, ^{13}C NMR, CHN and ESI-MS, which matched reasonably well with the reported data [27]. The 1,3-dipolar nitrone cycloaddition was found regioselective in the presence of bolaamphiphile **5c** as catalyst. Like previous cases, 5-substituted isoxazolidines (**9**) were favored over 4-substituted regiomers (**8**). In case of *o*-nitrobenzaldehyde only 5-substituted isoxazolidines (**9a**) were obtained. It was also observed that 4-substituted isomers (**8**) form as the minor product (only 10–15%) from aldehyde with EWG (Table 2, entries 1–3, 6, 7), whereas, the amount of **8** significantly increases (up to 30%) in the product mixture for the aldehydes with EDG (Table 2, entries 4, 5). For compounds **8**(**b–g**) endo isomers were formed as the sole product and no trace of exo isomer was found in the reaction mixture (Table 2, entries 2–7). Only in case of
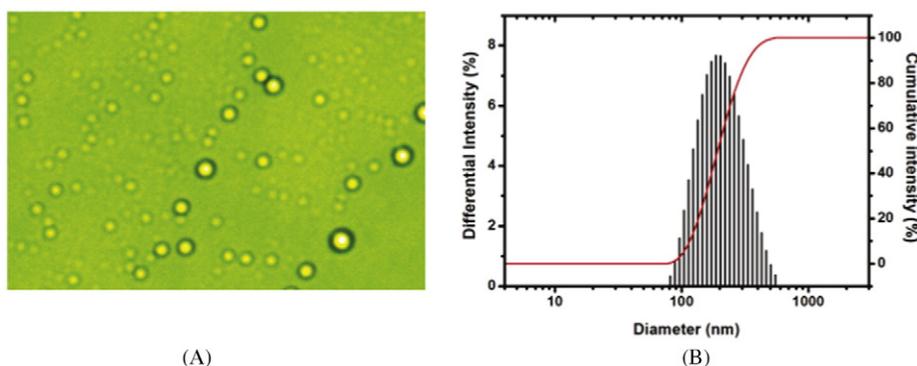

Fig. 2. (A) A typical optical micrograph of emulsion droplets formed in an aqueous solution of bolaamphiphile (**5c**), *m*-nitrobenzaldehyde and phenylhydroxylamine; (B) DLS data of bolaamphiphile **5c** containing solution showing formation of aggregates.

Table 1
Selection of suitable catalyst and reaction condition.

Entry	ArCHO	Catalyst	Mol% of catalyst	Time (h)	%Yield[a] (8:9)	8 (exo:endo)	9 (cis:trans)
1	m—NO$_2$C$_6$H$_4$—	5a	2	12	80 (1:8)	endo only	1:2
2	m—NO$_2$C$_6$H$_4$—	5b	2	10	76 (1:6)	endo only	1:2
3	m—NO$_2$C$_6$H$_4$—	5c	2	12	84 (1:6)	endo only	2:5
4	m—NO$_2$C$_6$H$_4$—	5c	5	10	85 (1:6)	endo only	2:5
5	m—NO$_2$C$_6$H$_4$—	5c	10	10	82 (1:6)	endo only	2:5
6	m—NO$_2$C$_6$H$_4$—	SDS	10	30	81	–	4:5
11	p—OMeC$_6$H$_4$—	5a	2	36	78 (2:7)	endo only	4:5
12	p—OMeC$_6$H$_4$—	5b	2	30	82 (2:3)	endo only	1:1
13	p—OMeC$_6$H$_4$—	5c	2	30	85 (1:2)	endo only	2:3
14	p—OMeC$_6$H$_4$—	SDS	10	76	84	–	cis only

  [a]   **8** and **9** are isolated separately and the yields presented in Table 1 is the combined yield of them.

cinnamaldehyde negligible diastereoselectivity was observed (Table 2, entry 8). The formation of endo isomer was established by a smaller coupling constant value between H$_3$–H$_4$ (J_{3-4} = 5.5–6 Hz) [27]. For 5-substituted isomers (**9**) moderate distereoselectivity was observed except for 2,5-methoxybenzaldehyde for which only trans product was formed (Table 2, entry 5). The diastereomeric ratio in **9** was determined in a qualitative manner from the integration values of separate peaks of cis and trans isomers in ^{1}H NMR and ^{13}C NMR spectra of the inseparable mixture [27,35]. It is noteworthy to mention that the yield and diastereoselectivity were poor in case of 2,5-methoxybenzaldehyde as substrate for SDS catalyzed reaction as reported by Chatterjee et al. [27]. However, it may be considered that the inherent chirality in the catalyst (**5c**) is not properly translated to the transition states of the reaction intermediates which is the consequence of inconsistent regio- and stereo-selectivity.

One crucial aspect of a catalytic transformation is the reusability of the catalyst without much change in yield and stereoselectivity. This is a value addition to any process particularly from industrial perspective. As the products were easily taken up in the organic layer, water layer containing catalyst **5c** was reused for five consecutive cycles preserving diastereoselectivity and without observable change in the yields of the final products (for details see SI, Table S1).

Table 2
Bolaamphiphile (**5c**) catalyzed water exclusion reaction.

Entry	ArCHO Ar = (a–h)	Time (h)	%Yield (8:9)[a]	8 (exo:endo)	9 (cis:trans)
1	a = o—NO$_2$C$_6$H$_4$—	12	82	–	2:3
2	b = m—NO$_2$C$_6$H$_4$—	12	84 (1:6)	endo only	2:5
3	c = p—NO$_2$C$_6$H$_4$—	12	92 (1:8)	endo only	3:4
4	d = p—OMeC$_6$H$_4$—	39	85 (1:2)	endo only	4:5
5	e = 2.5-diOMe—C$_6$H$_3$—	15	81 (1:2)	endo only	trans only
6	f = p—ClC$_6$H$_4$—	20	79 (1:6)	endo only	3:1
7	g = p—BrC$_6$H$_4$—	24	87 (1:6)	endo only	4:5
8	h = C$_6$H$_5$CH=CH—	30	93 (2:3)	5:4	5:4

  [a]   **8** and **9** are isolated separately and the yields presented in Table 1 is the combined yield of them.

3. Conclusion

In conclusion, a simple and efficient route has been developed for the synthesis of a new series of a novel class of D-glucose based anionic bolaamphiphiles using the C-3 functionality of diisopropylidene glucose. They showed low CMC and interesting morphologies when dispersed in aqueous solution. These chiral anionic bolaamphiphiles were efficiently used to catalyze dehydrative 1,3-dipolar nitrone cycloaddition with just 2 mol% of catalyst loading. The corresponding isoxazolidine derivatives were formed with moderate to high regio- and stereo-selectivity. The bolaamphiphile with 12 carbon spacer (**5c**) was found to be a better candidate among the rest. The catalyst was successfully reused for five consecutive cycles without much change in activity.

Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.catcom.2017.02.021.

References

[1] J.-H. Fuhrhop, T. Wang, Bolaamphiphiles, Chem. Rev. 104 (2004) 2901–2937.
[2] R. Zana, I.D. Robb (Eds.), In Specialist Surfactants, Chapman & Hall, Glasgow 1996, p. 81.
[3] N. Nuraje, H. Bai, K. Su, Bolaamphiphilic molecules: assembly and applications, Prog. Polym. Sci. 38 (2013) 302–343.
[4] R. Iwaura, K. Yoshida, M. Masuda, K. Yase, T. Shimizu, Spontaneous fiber formation and hydrogelation of nucleotide bolaamphiphiles, Chem. Mater. 14 (2002) 3047–3053.
[5] H. Zeng, M.E. Johnson, N.J. Oldenhuis, T.N. Tiambeng, Z. Guan, Structure-based design of dendritic peptide bolaamphiphiles for siRNA dfelivery, ACS Cent. Sci. 1 (2015) 301–312.

[6] S. Sistach, K. Rahme, N. Pérignon, J.-D. Marty, N.L.-D. Viguerie, F. Gauffre, C. Mingotaud, Bolaamphiphile surfactants as nanoparticle stabilizers: Application to reversible aggregation of gold nanoparticles, Chem. Mater. 20 (2008) 1221–1223.

[7] J.-N. Tian, B.-Q. Ge, Y.-F. Shen, Y.-X. He, Z.-X. Chen, Thermodynamics and structural evolution during a reversible vesicle — micelle transition of a vitamin-derived bolaamphiphile induced by sodium cholate, J. Agric. Food Chem. 64 (2016) 1977–1988.

[8] R. Iwaura, K. Yoshida, M. Masuda, M. Ohnishi-Kameyama, M. Yoshida, T. Shimizu, Oligonucleotide-templated self-assembly of nucleotide bolaamphiphiles: DNA-like nanofibers edged by a double-helical arrangement of A–T base pairs, Angew. Chem. Int. Ed. 42 (2003) 1009–1012.

[9] Y. Jin, R. Xin, L. Tong, L. Du, M. Li, Combination anti-HIV therapy with the self-assemblies of an asymmetric bolaamphiphilic zidovudine/didanosine prodrug, Mol. Pharm. 8 (2011) 867–876.

[10] S. Schmid, D.Y.W. Ng, E. Mena-Osteritz, Y. Wu, T. Weil, P. Bäuerle, Self-assembling oligothiophene–bolaamphiphiles for loading and controlled release of doxorubicin into living cells, Chem. Commun. 52 (2016) 3235–3238.

[11] M. Brunelle, A. Polidori, S. Denoyelle, A.-S. Fabiano, P.Y. Vuillaume, S. Laurent-Lewandowski, B. Pucci, A structure activity investigation of hemifluorinated bifunctional bolaamphiphiles designed for gene delivery, C. R. Chim. 12 (2009) 188–208.

[12] G. Yu, J. Li, W. Yu, C. Han, Z. Mao, C. Gao, F. Huang, Carbon nanotube/biocompatible bola-amphiphile supramolecular biohybrid materials: preparation and their application in bacterial cell agglutination, Adv. Mater. 25 (2013) 6373–6379.

[13] M. Berchel, L. Lemiègre, S. Trépout, O. Lambert, J. Jeftić, T. Benvegnu, Synthesis of unsymmetrical saturated or diacetylenic cationic bolaamphiphiles, Tetrahedron Lett. 49 (2008) 7419–7422.

[14] M. Gubitosi, L. Travaglini, A. D'Annibale, N.V. Pavel, J.V. Tato, M. Obiols-Rabasa, S. Sennato, U. Olsson, K. Schillén, L. Galantini, Sugar — bile acid-based bolaamphiphiles: from scrolls to monodisperse single-walled tubules, Langmuir 30 (2014) 6358–6366.

[15] G.L. Sorella, G. Strukul, A. Scarso, Recent advances in catalysis in micellar media, Green Chem. 17 (2015) 644–683.

[16] G.B. Mullen, T.R. Decory, J.T. Mitchell, S.D. Allen, C.R. Kinssolving, V.S. Georgiev, Studies on antifungal agents. Novel substituted 3,5-diphenyl-3-(1*H*-imidazol-1-ylmethy1)-2-a1kylisoxazolidine derivatives, J. Med. Chem. 31 (1988) 2008–2014.

[17] Raunak V. Kumar, S. Mukherjee, A.K. Poonam, K. Prasad, C.E. Olsen, S.J.C. Schaffer, S.K. Sharma, A.C. Watterson, W. Errington, V.S. Parmar, Microwave mediated synthesis of spiro-(indoline-isoxazolidines): mechanistic study and biological activity evaluation, Tetrahedron 61 (2005) 5687–5697.

[18] Y. Wu, G.F. Dai, J.H. Yang, Y.X. Zhang, Y. Zhu, J.C. Tao, Stereoselective synthesis of 15- and 16-substituted isosteviol derivatives and their cytotoxic activities, Bioorg. Med. Chem. Lett. 19 (2009) 1818–1821.

[19] A. Rescifina, M.A. Chiacchio, A. Corsaro, E.D. Clercq, D. Iannazzo, A. Mastino, A. Piperno, G. Romeo, R. Romeo, V. Valveri, Synthesis and biological activity of isoxazolidinyl polycyclic aromatic hydrocarbons: potential DNA intercalators, J. Med. Chem. 49 (2006) 709–715.

[20] I.A. Grigoŕev, in: H. Feuer (Ed.), In nitrile oxides, nitrones, and nitronates in organic synthesis, second ed.John Wiley and Sons, Hoboken, New Jersey 2008, pp. 129–434.

[21] M.S. Singh, S. Chowdhury, S. Koley, Progress in 1,3-dipolar cycloadditions in the recent decade: an update to strategic development towards the arsenal of organic synthesis, Tetrahedron 72 (2016) 1603–1644.

[22] R. Jasiński, M. Ziółkowska, O.M. Demchuk, A. Maziarka, Regio- and stereoselectivity of polar [2 + 3] cycloaddition reactions between (Z)-C-(3,4,5-trimethoxyphenyl)-N-methylnitrone and selected (E)-2-substituted nitroethenes, Cent. Eur. J. Chem. 12 (2014) 586–593.

[23] R. Jasinski, A stepwise, zwitterionic mechanism for the 1,3-dipolar cycloaddition between (Z)-C-4-methoxyphenyl-N-phenylnitrone and *gem*-chloronitroethene catalyzed by 1-butyl-3-methylimidazolium ionic liquid cations, Tetrahedron Lett. 56 (2015) 532–535.

[24] T. Mita, N. Ohtsuki, T. Ikeno, T. Yamada, Enantioselective 1,3-dipolar cycloaddition of nitrones catalyzed by optically active cationic cobalt(III) complexes, Org. Lett. 4 (2002) 2457–2460.

[25] C. Lu, A.V. Dubrovskiy, R.C. Larock, Synthesis of benzisoxazolines by the coupling of arynes with nitrones, J. Org. Chem. 77 (2012) 2279–2284.

[26] E. Falkowska, M.Y. Laurent, V. Tognetti, L. Joubert, P. Jubault, J.-P. Bouillon, X. Pannecoucke, Synthesis of SF_5-substituted isoxazolidines using 1,3-dipolar cycloaddition reactions of nitrones with pentafluorosulfanyl acrylic esters and amides, Tetrahedron 71 (2015) 8067–8076.

[27] A. Chatterjee, D.K. Maiti, P.K. Bhattacharya, Water exclusion reaction in aqueous media: nitrone formation and cycloaddition in a single pot, Org. Lett. 5 (2003) 3967–3969.

[28] B. Chakraborty, G.P. Luitel, An efficient ecofriendly protocol for the synthesis of novel fluoro isoxazoline and isoxazolidines using N-benzyl fluoro nitrone via cycloaddition reactions, Tetrahedron Lett. 54 (2013) 765–770.

[29] O. Bortolini, I. Mulani, A.D. Nino, L. Maiuolo, M. Nardi, B. Russo, S. Avnet, Efficient synthesis of isoxazolidine-substituted bisphosphonates by 1,3-dipolar cycloaddition reactions, Tetrahedron 67 (2011) 5635–5641.

[30] Z.T. Bhutia, P. Geethika, A. Malik, V. Kumar, A. Chatterjee, B.G. Roy, M. Banerjee, In situ mechanochemical synthesis of nitrones followed by 1,3-dipolar cycloaddition: a catalystfree, "green" route to cis-fused chromano[4,3-c] isoxazoles, RSC Adv. 5 (2015) 99566–99572.

[31] V. Kumar, A. Chatterjee, N. Kumar, A. Ganguly, I. Chakraborty, M. Banerjee, D-Glucose derived novel gemini surfactants: synthesis and study of their surface properties, interaction with DNA, and cytotoxicity, Carbohydr. Res. 397 (2014) 37–45.

[32] Y. Yan, T. Lu, J. Huang, Recent advances in the mixed systems of bolaamphiphiles and oppositely charged conventional surfactants, J. Colloid Interface Sci. 337 (2009) 1–10.

[33] L. Lakhrissi, N. Hassan, B. Lakhrissi, M. Massoui, E.M. Essassi, J.M. Ruso, C. Solans, C. Rodriguez-Abreu, Surfactants based on bis-galactobenzimidazolones: synthesis, self-assembly and ion sensing properties, J. Surfactant Deterg. 14 (2011) 487–495.

[34] The TEM facility has been outsourced from SICART-CVM, Anand, Gujarat, India and method is obtained from Mr. Vikas A. Patel, Technical Assistant, SICART-CVM, Gujarat, India.

[35] D.A.L. Otte, D.E. Borchmann, C. Lin, M. Weck, K.A. Woerpel, ^{13}C NMR spectroscopy for the quantitative determination of compound ratios and polymer end groups, Org. Lett. 16 (2014) 1566–1569.