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Ultrasound Assisted One-Pot Synthesis of Novel 3-(Aryl)-5-((4-(phenyldiazenyl)phenoxy)methyl)isoxazolines in Water

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Abstract: In this work, we present an efficient one-pot method for the synthesis of three new azo-isoxazoline derivatives (**4a–c**) from aromatic aldehydes, hydroxylamine hydrochloride and 4-(allyloxy)azobenzene. Thus, the azo-isoxazoline derivatives (**4a–c**) were synthesized via 1,3-dipolar cycloaddition using sodium dichloroisocyanurate (SDIC) as an eco-friendly and inexpensive oxidizing agent under ultrasound cavitation in water as a green solvent. The desired compounds **4a–c** were obtained in high to excellent yields of 75–90%.

Keywords: three-component reactions; one-pot approach; 1,3-dipolar cycloaddition; ultrasound cavitation; azo-isoxazolines

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1. Introduction

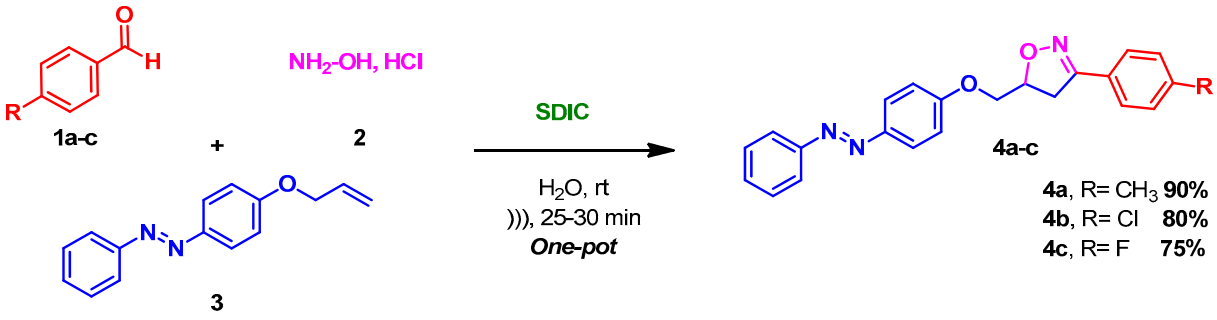
Azo dyes belong to the most important class of chromophores, which are characterized by nitrogen–nitrogen double bonds «N=N», and have a wide range of applications in industry and pharmaceutical sectors [1,2]. In addition, azo dyes containing heterocycles have a wide spectrum of biological activities, which is mostly influenced by the nature of the heterocycle and position of the substituents, such as antibacterial, antifungal, antiviral, antitubercular, anticancer, anticonvulsant, antidiabetic, analgesic and anti-inflammatory, and chemosensing activities [3,4], hence the interest to synthesize more heterocycles with azo dyes.

On the other hand, isoxazolines are an important class of heterocyclic compounds that contain nitrogen and oxygen heteroatoms, which are very important in medicinal chemistry due to their various pharmacological properties, including anti-diabetic [5], anti-cancer [6], anti-inflammatory [7], antimicrobial [8], anti-stress [9], anti-Alzheimer [10], analgesic [11] and insecticidal properties [12].

Several approaches and methods for the synthesis of isoxazoline rings have been reported in the literature [13–18]. However, most 1,3-dipolar cycloadditions of alkenes with nitrile oxides are produced by the oxidation of aldoximes in organic solvents under conventional conditions, including metal-catalyzed processes [14–18]. Numerous oxidants, including $Ni(ClO_4)_2$ $6H_2O$ and $NiBF_4$ $6H_2O$ as Lewis acid catalysts [14], Ru(acetone)(R,R)-(BIPHOP-F)Cp][SbF_6] [15], organo-hypervalent iodine reagents [16], CrO_2 and MnO_2 [17], have been used to generate nitrile oxides from aldoximes. Other examples of the use of rare earth elements as catalysts in 1,3-dipolar cycloaddition reactions of nitrile oxide have also been described [18].

However, in the context of the current green chemistry trend, the use of alternative, non-toxic and renewable reaction media is attractive, such as water [19,20] and ionic liquids (ILs) [21], as well as alternative activation conditions, such as microwave (MW) [22,23] and ultrasound (US) activation [24,25].

As a part of our research into the use of selective, environmentally friendly and efficient catalysts combined with ultrasound as an alternative source of energy for the synthesis of bioactive aza-heterocycles [22,25–30], herein, we report a one-pot three-component approach for the synthesis of novel azo-isoxazolines (**4a–c**) from aromatic aldehydes **1a–c**, hydroxylamine hydrochloride **2**, and 4-(allyloxy)azobenzene **3**, catalyzed by SDIC (or NaDCC) in water under ultrasound cavitation at room temperature (Scheme 1).



Scheme 1. Synthesis of 3-(aryl)-5-((4-(phenyldiazenyl)phenoxy)methyl)isoxazolines **4a–c**.

2. Results and Discussion

Recently, we reported a simple and environmentally friendly method for the synthesis of 3,5-disubstituted isoxazoles under ultrasonic cavitation using SDIC as an eco-friendly and inexpensive oxidant in water [25]. It is important to extend the application of this method to the synthesis of other heterocycles. First, our investigations were initiated with the condensation of aromatic aldehydes **1a–c** with hydroxylamine, easily generated from hydroxylamine hydrochloride **2** under basic conditions, to provide the appropriate nitrile oxide in situ by oxidation of aromatic aldoximes by SDIC in water under ultrasonic cavitation. Thus, the nitrile oxide was trapped by 4-(allyloxy)azobenzene **3** as a dipolarophile via 1,3-dipolar cycloaddition, to give novel azo-isoxazolines **4a–c**. The reaction was completed in 25–30 min via ultrasound cavitation, as attested by TLC monitoring, in good to excellent yields of 75–90% (Scheme 1).

In order to examinate the scope and limitations of this ultrasound-assisted reaction, we evaluated the reactivity of three aldehydes **1a–c**. In general, we observed that both electron-poor and electron-rich aldehydes can be used in this reaction, leading to azo-isoxazolines isolated as pure products in good to excellent yields (75–90%), and high regioselectivity.

The azo-isoxazolines (**4a–c**) were fully characterized by ^{1}H, ^{13}C NMR, and LCMS spectra (See Supplementary Materials). For example, the ^{1}H NMR spectrum of 5-((4-(phenyldiazenyl)phenoxy)methyl)-3-(p-tolyl)-isoxazoline **4a** showed a multiplet at δ = 5.22–5.11 ppm due to the H-isoxazolinic proton, and two doublets of doublets at δ = 3.42 and 3.57 ppm for $C4_{HaHb}$ isoxazolinic protons. The N–CH_2 protons appear as two doublets of doublets at δ = 4.18 and 4.28 ppm. This compound also showed a singlet, on average at δ = 2.42 ppm, which identifies the methyl group; thus, the presence of the signals at δ = 7.06–7.99 ppm is attributable to the different aromatic protons. The ^{13}C NMR spectrum of **4a** exhibited characteristic signals at δ = 21.5 ppm (CH_3), 37.8 ppm (CH_2-isoxazoline), 68.9 ppm (N-CH_2), 78.4 ppm (CH-isoxazoline), 156.40 ppm (C=N-isoxazoline), and 160.97 ppm, 152.51 ppm, 147.2 ppm, 140.6 ppm, 130.6 ppm, 129.5 (2C) ppm, 129.1 (2C) ppm, 126.8 (2C) ppm, 126.4 ppm, 124.9 (2C) ppm, 122.6 (2C) ppm, and 114.9 (2C) ppm, attributable to aromatic carbons.

3. Materials and Methods

All organic solvents and chemicals were bought from commercial sources (Merck, Sigma-Aldrich, St. Louis, MO, USA). TLC was used to monitor the reaction on silica gel 60 F254 plates, visualized via UV light. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC 400 spectrometer in $CDCl_3$. High-resolution mass spectra (HRMS) were obtained with a LTQ Orbitrap hybrid mass spectrometer with an electrospray ionization probe (Thermo Scientific, San Jose, CA, USA) by direct infusion from a pump syringe to confirm the correct molar mass and high purity of compounds. The Munz Köfler Bench System was used to measure the melting points. An "Elmasonic S 30/S 30 H UltraSonic Bath Cleaner" with an effective ultrasonic power of 80 W and a frequency of 47 kHz was used to perform the ultrasound-assisted reactions.

3-(Aryl)-5-((4-(phenyldiazenyl)phenoxy)methyl)isoxazoline **4a**–**c**: To a solution of aromatic aldehyde **1a**–**c** (1 mmol) and hydroxylamine hydrochloride **2** (1.2 mmol) in H_2O (10 mL), SDIC (0.5 mmol) was added. The reaction mixture was sonicated for 10 min at 25 °C (TLC monitoring). 4-(Allyloxy)azobenzene **3** was then added sequentially, and the mixture was sonicated using an ultrasonic bath (47 kHz) for 15 to 20 min (TLC monitoring). The mixture was extracted using CH_2Cl_2 (2 × 15 mL) and washed with saturated brine solution (15 mL × 2) and water (20 mL), dried over Na_2SO_4, and concentrated in vacuum. To obtain the pure azo-isoxazolines **4a**–**c**, the residue was purified by recrystallization in EtOH, by dissolving the crude product in heated ethanol (10 mL). Then, the mixture was cooled down and the pure azo-isoxazolines **4a**–**c** were isolated by filtration.

3-(4-Methylphenyl)-5-((4-(phenyldiazenyl)phenoxy)methyl)-isoxazoline (**4a**): Yellow solid, yield 90%, m.p. 124–125 °C, TLC (cyclohexane/AcOEt, 5/5, *v*/*v*) R_f = 0.48; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99–7.95 (m, 2H, H_{Ar}), 7.94–7.91 (m, 2H, H_{Ar}), 7.63 (d, *J* = 8.2 Hz, 2H, H_{Ar}), 7.56–7.50 (m, 2H, H_{Ar}), 7.49–7.45 (m, 1H, H_{Ar}), 7.28–7.24 (m, 2H, H_{Ar}), 7.06 (d, *J* = 9.0 Hz, 2H, H_{Ar}), 5.22–5.11 (m, 1H, C_5H isoxazoline), 4.28 (dd, *J* = 9.8, 5.0 Hz, 1H, N-CH), 4.18 (dd, *J* = 9.9, 5.6 Hz, 1H, N-CH), 3.57 (dd, *J* = 16.7, 10.7 Hz, 1H, C_4H isoxazoline), 3.42 (dd, *J* = 16.7, 6.9 Hz, 1H, C_4H isoxazoline), 2.42 (s, 3H, CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.9, 156.4, 152.5, 147.2, 140.6, 130.6, 129.5 (2C), 129.1 (2C), 126.8 (2C), 126.4, 124.9 (2C), 122.6 (2C), 114.9 (2C), 78.4, 68.9, 37.8, 21.5; HRMS: Calcd. for $C_{23}H_{21}N_3O_2H$+ ($[M + H]^+$): 372.1634, Found: 372.1692.

3-(4-Chlorophenyl)-5-((4-(phenyldiazenyl)phenoxy)methyl)-isoxazoline (**4b**): Yellow solid, yield 80%, m.p. 130–132 °C, TLC (cyclohexane/AcOEt, 5/5, *v*/*v*) R_f = 0.5; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 9.0 Hz, 2H, H_{Ar}), 7.93–7.89 (m, 2H, H_{Ar}), 7.67 (d, *J* = 8.6 Hz, 2H, H_{Ar}), 7.56–7.50 (m, 2H, H_{Ar}), 7.48 (d, *J* = 7.3 Hz, 1H, H_{Ar}), 7.43 (d, *J* = 8.6 Hz, 2H, H_{Ar}), 7.05 (d, *J* = 9.0 Hz, 2H, H_{Ar}), 5.24–5.15 (m, 1H, C_5H isoxazoline), 4.29 (dd, *J* = 9.9, 4.8 Hz, 1H, N-CH), 4.19 (dd, *J* = 9.9, 5.4 Hz, 1H, N-CH), 3.55 (dd, *J* = 16.8, 10.8 Hz, 1H, C_4H isoxazoline), 3.42 (dd, *J* = 16.7, 7.1 Hz, 1H, C_4H isoxazoline). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.8, 155.3, 152.6, 147.1, 136.0, 130.6, 129.1 (2C), 129.1 (2C), 128.0 (2C), 127.8, 124.8 (2C), 122.6 (2C), 114.9 (2C), 78.9, 68.7, 37.4; HRMS: Calcd. for $C_{22}H_{18}ClN_3O_2H$+: ($[M + H]^+$): 392.1088, Found: 392.1142.

3-(4-Fluorophenyl)-5-((4-(phenyldiazenyl)phenoxy)methyl)-isoxazoline (**4c**): Yellow solid, yield 75%, m.p. 135–137 °C, TLC (cyclohexane/AcOEt, 6/4, *v*/*v*) Rf = 0.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 9.0 Hz, 2H, H_{Ar}), 7.93–7.88 (m, 2H, H_{Ar}), 7.76–7.69 (m, 2H, H_{Ar}), 7.56–7.50 (m, 2H, H_{Ar}), 7.49–7.44 (m, 1H, H_{Ar}), 7.18–7.11 (m, 2H, H_{Ar}), 7.05 (d, J = 9.0 Hz, 2H, H_{Ar}), 5.23–5.14 (m, 1H, C_5H isoxazoline), 4.28 (dd, J = 9.9, 4.9 Hz, 1H, N-CH), 4.18 (dd, J = 9.9, 5.6 Hz, 1H, N-CH), 3.56 (dd, J = 16.7, 10.7 Hz, 1H, C_4H isoxazoline), 3.42 (dd, J = 16.7, 7.0 Hz, 1H, C_4H isoxazoline). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.89 (d, J = 250.9 Hz), 160.8, 155.5, 152.6, 147.4, 130.6, 129.1 (2C), 128.8, 128.7, 125.5 (d, J = 3.6 Hz), 124.8 (2C), 122.6 (2C), 116.1, 115.9, 114.9 (2C), 78.7, 68.7, 37.7.; HRMS: Calcd. for $C_{22}H_{18}FN_3O_{2H}$+: ($[M + H]^+$): 376.1383, Found: 376.1440.

4. Conclusions

In conclusion, a simple and green one-pot three-component approach was developed for the synthesis of three novel azo-isoxazoline derivatives from aromatic aldehydes, hydroxylamine hydrochloride, and 4-(allyloxy)azobenzene. The benefits of this approach include the use of readily available raw materials, atom economy, a green solvent, process simplification, and straightforward target product isolation. In addition, the chemical structures of azo-isoxazolines (**4a–c**) were confirmed using spectroscopy techniques such as ^{1}H NMR, ^{13}C NMR, and HRMS.

Supplementary Materials: The following supporting information can be downloaded online. Figure S1: ^{1}H NMR spectrum of **4a**; Figure S2: ^{13}C NMR spectrum of **4a**; Figure S3: ^{1}H NMR spectrum of **4b**; Figure S4: ^{13}C NMR spectrum of **4b**; Figure S5: ^{1}H NMR spectrum of **4c**; Figure S6: ^{13}C NMR spectrum of **4c**; Figure S7: HRMS spectrum of **4a**; Figure S8: HRMS spectrum of **4b**; Figure S9: HRMS spectrum of **4c**.

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Sample Availability: Samples of the compounds are available from the authors.

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