Facile sonochemical heterocyclization of 2,5-dimethoxy tetrahydrofuran with primary amines using sulfonated MWCNTs as a recyclable catalyst in aqueous media

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ABSTRACT
A set of N-substituted pyrrole derivatives have been designed and synthesized using sulfonated multi-walled carbon nanotubes as a recyclable heterogeneous catalyst under ultrasound irradiation. This reaction was carried out between 2,5-dimethoxy tetrahydrofuran and primary amines in water under green conditions. This method has some advantages such as: short reaction times, excellent product yields, simplicity of the procedure, easy work-up and high purity of products.

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Introduction

Pyrroles are among the most important heterocyclic compounds with diverse biological activities and pharmaceutical agents (1). They are found as the fundamental structural origin in various classes of natural and biologically important compounds such as coenzymes, porphyrins, bile pigments and alkaloids (2–7). They are also precious intermediates in organic reactions (8). Consequently, a wide range of methods have been devised for the synthesis of pyrroles. However, many of the methods are included such as: Paal–Knorr reaction (9,10), aza-Michael addition (11) and multicomponent reactions (12). In recent years, the synthesis of pyrrole derivatives has been reported in the presence of different catalysts such as: Bi(NO₃)₃·5H₂O/MW (13), molecular iodine/MW (14) and PMA/SiO₂ (15), Fe₂O₃·SO₃H on Fe₃O₄/MW (16), H₃PW₁₂O₄₀ on SiO₂/MW (17), nano FGT/H₂O/MW (18,19), γ-Fe₂O₃·SiO₂-Sb-IL/Reflux (20), Bi(NO₃)₃·5H₂O/US (21).

Although these reported works are convenient methods for the preparation of pyrroles, the present method compared with the used catalysts has some advantages such as thermal and chemical stability, non-flammability properties, high activity, simplicity of the procedure, easy reaction work-up, recyclability, cleans method and resulting products with high purity.

Most of the catalysts contain metals or metal complexes supported on a material with suitable surface area as support. The most important characteristics of a support are the surface area, chemical composition, electronic, mechanical and stability properties. Until now, many allotropes of carbon have been discovered such as graphene, fullerene and carbon nanotubes (CNTs). They are used as support for the synthesis of heterogeneous catalyst. Chemical modification of CNTs is a method for enhancing their usages such as catalytic applications (22), medicine (23), optical devices (24) and electronic instruments. These applications made them
a significant material in science and technology. Chemical oxidation of nanotubes is generally carried out using gaseous or liquid oxidants. A more general procedure used for CNTs oxidation is that of using oxidizing agents in aqueous solutions. Oxidation of MWCNTs using acid-sonication has been studied by Xing et al., who demonstrated that the dispersion stability and solubility of the treated MWCNTs in water improve significantly (25). Also, the ozone in the presence of an oxidative ozonide cleavage reagent (H₂O₂) has been reported as a good oxidizing agent (26).

The application of ultrasonic irradiation in organic reactions has made progress in recent years. Sonication increases the reaction rate and eschews the use of high reaction temperatures (27). The advantages of the ultrasound-assisted method such as short reaction times, good yields and mild reaction conditions are well documented (28). The effects of ultrasonic irradiation obtained during organic reactions are due to cavitations. In the case of volatile component holes are trusty to act as micro reactor; as the volatile components enter the micro bubbles, high temperature and pressure are produced during breaking of their chemical bonds hence reacting with other species (29–31). The presence of ultrasound irradiation in the liquid–liquid system, cavitational falling near the liquid–liquid juncture interrupts the interface and induce of one liquid into the other formed emulsion, and leads to a dramatic increase in the interfacial contact area across which transfer of species can take place.

In continuation of our recent work on using the ultrasound in organic synthesis (32–35) and catalytic reaction (36), herein we hope to report an efficient ultrasound-assisted eco-friendly method for the synthesis of N-substituted pyrroles by treatment of 2,5-dimethoxy tetrahydrofuran and various primary aromatic amines in the presence of sulfonated multi-walled CNTs as a heterogeneous catalyst in aqueous media.

Results and discussion

Preparation and characterization of the catalyst

The preparation of the MWCNTs-SO₃H catalyst was performed in two steps as described in Scheme 1. In the first step, MWCNTs were converted to MWCNTs-COOH with HCl (37%) and HNO₃ (63%). This step was carried out in order to purify of MWCNTs and to remove fullerene, amorphous carbon and metal particles such as Fe, Co, Ni and increase the reactivity (Scheme 1). This step also introduced oxygen comprising groups, mainly carboxyl groups on the MWCNTs (37,38). After this step, sulfuric acid (98%) was added to the MWCNTs-COOH to obtain the MWCNTs-SO₃H. The prepared catalyst was revealed by scanning electron microscopy (SEM), X-ray

![Scheme 1](image1.png)

**Scheme 1.** Preparation of the MWCNTs-SO₃H catalyst.

![Scheme 2](image2.png)

**Scheme 2.** Reaction of 2,5-dimethoxy tetrahydrofuran and primary aromatic amines.
powder diffraction (XRD), FT-IR, BET, electron dispersive X-ray (EDX) and X-ray photoelectron spectroscopy (XPS) spectroscopy (Figures 1S−6S and Table 1S in the Supporting Information) (39,40).

The reaction of 2,5-dimethoxy tetrahydrofuran with different types of primary aromatic amines in 1.2:1 mole ratio has been studied in the presence of MWCNT-SO₃H as the heterogeneous catalyst under ultrasonic irradiation at a power of 65 W in water as a solvent (Scheme 2).

In the first step, the optimization of the catalyst amount in the reaction was carried out. The reaction of 2,5-dimethoxy tetrahydrofuran with 4-methoxy aniline with 1.2:1 mol ratio was done with different amounts of catalyst under ultrasonic irradiation at a power of 60 W and water as the solvent. The obtained results are presented in Table 1. Taking into consideration the results in this table, the optimum amount of used MWCNT-SO₃H as the catalyst in this reaction was obtained: 0.02 g (Table 1, entry 2). After the optimization of the catalyst amount, the reaction was carried out in water under ultrasonic irradiation at different powers (Table 2). In the same way, the highest yield for N-aryl pyrrole as product was resulted from the reaction run under ultrasonic irradiation with a power of 65 W (Table 2, entry 2).

Table 1. Optimization of the catalyst amounts under ultrasonic irradiation (60 W) in water.a.

| Entry | Amounts of catalyst (g) | Time (min) | Yield (%) |
|-------|-------------------------|------------|-----------|
| 1     | 0.01                    | 5          | 55        |
| 2     | 0.02                    | 5          | 85        |
| 3     | 0.03                    | 5          | 85        |

React condition: 2,5-dimethoxy tetrahydrofuran (1.2 mol), 4-methoxy aniline (1 mol), H₂O, ultrasound irradiation (60W), at room temperature, ultrasound time, 5 min.

Table 2. Optimization of ultrasonic power by using MWCNT-SO₃H (0.02 g) in water.a.

| Entry | Power (W) | Time (min) | Yield (%) |
|-------|-----------|------------|-----------|
| 1     | 60        | 5          | 85        |
| 2     | 65        | 5          | 96        |
| 3     | 70        | 5          | 70        |

React condition: 2,5-dimethoxy tetrahydrofuran (1.2 mol), 4-methoxy aniline (1 mol), MWCNT-SO₃H (0.02 g), H₂O, at room temperature, ultrasound time, 5 min.

Table 3. Optimization of solvents in the presence of MWCNT-SO₃H (0.02 g) under ultrasonic irradiation (65 W).a.

| Entry | Solvent | Time (min) | Yieldb (%) |
|-------|---------|------------|------------|
| 1     | EtOH    | 5          | 5          |
| 2     | CH₃CN   | 5          | 50         |
| 3     | DMF     | 5          | 90         |
| 4     | H₂O     | 5          | 96         |

React condition: 2,5-dimethoxy tetrahydrofuran (1.2 mol), 4-methoxy aniline (1 mol), MWCNT-SO₃H (0.02 g), at room temperature, ultrasound irradiation (65 W), H₂O as solvent.

Table 4. Synthesis of various N-aryl pyrrole using the catalytic amount of MWCNT-SO₂H as catalyst under ultrasonic irradiation in water.b.

| Entry | Amine | Product | Time (min): Yield (%) |
|-------|-------|---------|-----------------------|
| 1     |       |         | 5:95                  |
| 2     |       |         | 5.5:95                |
| 3     |       |         | 5:96                  |
| 4     |       |         | 5.2:93                |
| 5     |       |         | 5.2:93                |
| 6     |       |         | 5.9:5                 |
| 7     |       |         | 5.9:5                 |
| 8     |       |         | 5.9:5                 |
| 9     |       |         | 5.9:5                 |
| 10    |       |         | 5.9:5                 |
| 11    |       |         | 5.9:5                 |
| 12    |       |         | 5.9:5                 |
| 13`   |       |         | 5.9:5                 |
| 14`   |       |         | 5.9:5                 |
| 15`   |       |         | 5.9:5                 |

React condition: 2,5-dimethoxy tetrahydrofuran (1.2 mol) with different types of primary aromatic amines (1 mol), MWCNT-SO₂H (0.02 g), at room temperature, ultrasound irradiation (65 W), H₂O as solvent.

bIsolated yields.

`The reaction of 2,5-hexandione and aniline catalyzed by 5 mg graphene oxide at room temperature (41).

`The reaction of 2,5-hexandione with substituted anilines at room temperature (42).
Table 5. Reaction of 2,5-dimethoxy tetrahydrofuran with several amines in different conditions.

| Entry | Amounts of catalyst (g) | Time (min) | Product | Condition       | Yield (%) |
|-------|-------------------------|------------|---------|-----------------|-----------|
| 1     | 0.02                    | 60         | 3c      | Room temperature thermal (65°C) | –         |
| 2     | 0.02                    | 40         | 3c      | thermal (65°C)  | –         |
| 3     | 0.04                    | 55         | 3c      | Thermal (65°C)  | 50        |
| 4     | 0.04                    | 60         | 3a      | thermal (67°C)  | 48        |
| 5     | 0.04                    | 62         | 3h      | Thermal (70°C)  | 60        |
| 6     | 0.02                    | 7          | 3h      | U.S.            | 93        |
| 7     | 0.02                    | 5          | 3a      | U.S.            | 95        |
| 8     | 0.02                    | 5          | 3c      | U.S.            | 96        |

*The comparison conventional heating with ultrasound-assisted method.

In continuation, the reaction of 2,5-dimethoxy tetrahydrofuran with 4-methoxy aniline (1.2:1 mol ratio) in the presence of MWCNT-SO₃H (0.02 g) and power of 65 W was carried out in various solvents (Table 3). The best results were obtained using water as a solvent (Table 3, entry 4). It seems that ethanol was not a convenient solvent for this reaction due to the reasons such as the difference between dipole moment and polarity of ethanol and other used solvents for further solubilization of the reaction intermediates. Also, further ability of water for hydrogen bonding formation with reaction intermediates was compared with ethanol.

In order to ascertain the limitations of this protocol, the reaction of 2,5-dimethoxy tetrahydrofuran with several primary aromatic amines was carried out according to the general experimental procedure. The corresponding products and their results are summarized in Table 4. As can be seen from this table, the products were obtained in excellent yields and short reaction times for all of the entries except entry 10, 3j product, due to the steric hindrance of chlorine atom in the ortho situation of the amino group nucleophile that led to a reduction of the yield of reaction. Also, the usage of MWCNT-SO₃H as a catalyst in the present work results in higher yields and efficiencies and lower times for the preparation of some N-substituted pyroles compared with previously reported works using graphene oxide catalyst (41) and catalyst-free (42), in which the products were obtained in lower yields and longer reaction times (Table 4, entries 13–15). These results can be related to advantages of the catalyst role in this reaction.

In order to investigate the effects of ultrasonic irradiation and to evaluate and compare conventional heating with the ultrasound-assisted method, we continued our efforts under different conditions. The results are listed in Table 5. The ultrasound could increase the rate of reaction and therefore reduce the energy consumption. The chemical and physical effects of ultrasound are primarily from sonic cavitation, which includes formation, growth and collapse of the hole. The drive force behind the increased efficiency of N-aryl pyrroles formation by ultrasound is due to the increase in the temperature due to the formation of hot spots; and due to increase in the reactant impact surface area through the cavitations’ event.

In this study, N-aryl pyrroles as products of an efficient method were prepared through the reaction of 2,5-dimethoxy tetrahydrofuran and various primary aromatic amines in the presence of heterogeneous catalytic amount of MWCNT-SO₃H (0.02 g) under ultrasonic irradiation and water as a convenient solvent.

For practical applications of heterogeneous catalysts, the life-time of the MWCNTs-SO₃H and its level of reusability are very important features. At the end of each reaction, the catalyst was isolated by filtration, washed exhaustively with chloroform and ethanol and dried at 100°C for 24 h before being used with fresh materials. The catalyst was reused for four runs; the yields were ranged from 95% to 90% (Figure 1).

The structures of products and MWCNTs-SO₃H were confirmed by spectroscopic and physical data such as: IR, ¹H NMR, ¹³C NMR, XRD, SEM, BET, EDX, Raman and XPS spectroscopy. The ¹H NMR spectra of 1-(3-nitropheryl)-1H-pyrole showed the signals around δ = 6.33–7.34 ppm that are assigned to protons of aromatic rings. The ¹³C NMR spectra of the above-mentioned compound showed the signals around δ = 110.0–150.0 ppm that are assigned to carbon atoms of the aromatic rings.

A plausible mechanism for the formation of N-aryl pyroles is presented in Scheme 3. As can be seen, in the first step, the acetal group of 2,5-dimethoxy tetrahydrofuran compound was protonated (a). Then, the nucleophilic attack of the hydroxyl group of the substrate to the carbon atom of acetal produces the intermediate (b). Next, b was protonated by MWCNTs-SO₃H and dehydrated and converted to carbonyl group d, then, d was protonated by MWCNTs-SO₃H, and the nucleophilic attack of the amino group to carbonyl e occurs. The f was protonated, cyclized and dehydrated to give N-aryl pyrrole as a desired product.
**Experimental**

**Materials and apparatus**

All commercially available reagents were used without further purification and purchased from Merck Chemical Company in high purity. The used solvents were purified by standard procedures. IR spectra were obtained as KBr pellets on a Perkin-Elmer 781 spectrophotometer and on an impact 400 Nicolet FTIR spectrophotometer. \(^1\)H NMR and \(^{13}\)C NMR were recorded in CDCl\(_3\) and DMSO-d\(_6\) solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as the internal reference. UV-Vis spectra were recorded with a Perkin-Elmer 550 spectrophotometer. 1HN M R pellets on a Perkin-Elmer 781 spectrophotometer and on standard procedures. IR spectra were obtained as KBr disks and used in the synthesis of N-aryl pyrroles.

**General procedure for the synthesis of MWCNTs-SO\(_3\)H**

MWCNTs (1 g) and deionized water (100 ml) were added to a beaker and was sonicated for 30 min. After the sonication, the solvent was removed in vacuum and the obtained MWCNTs were transferred to another flask including 25 ml HNO\(_3\) (63%); 25 ml HCl (37%) and stirred at 80°C for 4 h under a nitrogen atmosphere. Then the solution was filtered in vacuum and washed thoroughly with deionized water several times. The obtained solid was dried at 100°C for 12 h. After this step, the MWCNTs-SO\(_3\)H was obtained and used in the synthesis of N-aryl pyrroles.

**General procedure for the synthesis of N-aryl pyrrole**

To a mixture of 2,5-dimethoxy tetrahydrofuran (1.2 mmol) and different primary aromatic amines (1 mmol) in water, MWCNT-SO\(_3\)H (0.02 g) was added and reacted under ultrasonic irradiation (65 W). The progress of the reactions was monitored by TLC (ethyl acetate/n-hexane 6/4). The catalyst along with the solid product was collected by filtration from the reaction mixture. The filtrated solid was dissolved in chloroform, the catalyst separated from the reaction mixture and washed with chloroform. The solvent was removed in vacuum to give the crude product. Then, the crude product was recrystallized from ethanol, N-aryl pyrroles were obtained as pure products. The recovered catalyst (0.019 g) was reused in the reaction for four runs. All of the pure products were characterized by comparison of their physical and spectral data with authentic samples (43–53).

1-Phenyl-1H-pyrrole (3a), brown solid; m.p. = 55–57°C, (m.p. = 56–58°C) (43); UV-Vis (CH\(_2\)Cl\(_2\)) \(\lambda_{max}\): 310; IR (KBr) \(\nu\) (cm\(^{-1}\)): 3033 (C–H, sp\(^2\) stretch), 1497, 1597 (C=C, Ar), 746, 749 (C–H, sp\(^3\) OOP); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) (ppm): 6.37–6.38 (d, 2H, J = 8.4 Hz, 2CH), 7.06–7.07 (d, 2H, J = 4.0 Hz, 2CH), 7.33–7.41 (m, 5H, 5CH).

1-p-Tolyl-1H-pyrrole (3b), black solid; m.p. = 79–81°C, (m.p. = 80–82°C) (44); UV-Vis (CH\(_2\)Cl\(_2\)) \(\lambda_{max}\): 315; IR (KBr) \(\nu\) (cm\(^{-1}\)): 3050 (C–H, sp\(^3\) stretch), 2922 (C–H, sp\(^3\)), 1509, 1616 (C=C, Ar); \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm): 2.38 (s, 3H, CH\(_3\)), 6.34 (s, 2H, 2CH), 7.01 (s, 2H, 2CH), 7.23 (d, 2H, J = 8.0 Hz, 2CH), 7.27–7.29 (d, 2H, J = 8.0 Hz, 2CH).

1-(4-Methoxy phenyl)-1H-pyrrole (3c), black solid; m.p. = 87–89°C, (m.p. = 88–89°C) (45); UV-Vis (CH\(_2\)Cl\(_2\)) \(\lambda_{max}\): 325; IR (KBr) \(\nu\) (cm\(^{-1}\)): 3033 (C–H, sp\(^2\) stretch), 2926 (C–H, sp\(^3\)), 1597 (C=C, Ar), 1310 (C–O), 749 (C–H sp\(^2\) OOP); \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm): 3.85 (s, 3H, CH\(_3\)), 6.33 (s, 2H, 2CH), 6.95–6.97 (d, 2H, J = 8.4 Hz, 2CH), 7.01 (s, 2H, 2CH), 7.31–7.34 (d, 2H, J = 8.4 Hz, 2CH).

1-(3-Methoxy phenyl)-1H-pyrrole (3d), yellow solid; m.p. = 46–48°C, (b.p. = 98–100°C) (46); UV-Vis (CH\(_2\)Cl\(_2\)) \(\lambda_{max}\): 320; IR (KBr) \(\nu\) (cm\(^{-1}\)): 3000 (C–H, sp\(^3\)), 2850 (C–H sp\(^3\)), 1400–1600 (C=C, Ar), 1300 (C–O), 743 (C–H sp\(^2\) OOP); \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) (ppm): 3.86 (s, 3H, CH\(_3\)), 6.26 (s, 2H, 2CH), 6.80–6.82 (d, 1H, J = 8.0 Hz, CH), 6.95 (s, 1H, CH), 7.00–7.02 (d, 1H, J = 8.0 Hz, CH), 7.11 (s, 2H, 2CH), 7.32–7.36 (t, 1H, J = 8.4 Hz, CH).

1-(4-Hydroxy phenyl)-1H-pyrrole (3e), brown solid; m.p. = 111–116°C, (m.p. = 113–115°C) (47); UV-Vis (CH\(_2\)Cl\(_2\)) \(\lambda_{max}\): 325; IR (KBr) \(\nu\) (cm\(^{-1}\)): 3448 (O-H, stretch), 3135 (C–H, sp\(^3\)), 1519 (C=C, Ar), 1024 (C–O, stretch), 732 (C–H sp\(^2\) OOP); \(^1\)H NMR (DMSO-d\(_6\), 400 MHz) \(\delta\) (ppm): 6.19 (s, 2H, 2CH), 6.52 (s, 1H, OH), 6.80–6.82 (d, 2H, J = 8.8 Hz, 2CH), 7.16 (s, 2H, 2CH), 7.31–7.33 (d, 2H, J = 8.4 Hz, 2CH).
Scheme 3. Plausible reaction mechanism for the preparation of N-aryl pyrroles.
Conclusion

In this research, we described the synthesis of N-aryl pyroles using 2,5-dimethoxy tetrahydrofuran with different primary aromatic amines. This reaction was carried out in the presence of catalytic amount of MWCNT-SO₃H under ultrasonic irradiations. The corresponding products were obtained in excellent yields, high purity and short reaction times. The products were confirmed by physical and spectroscopic data such as: IR, ¹H NMR, ¹³C NMR spectroscopy.

Disclosure statement

No potential conflict of interest was reported by the authors.

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