

Synthesis of New N-phenyl Fulleroisoxazoline in the Presence of Fe₃O₄@SiO₂ Nanoparticles as an Efficient Magnetically Recoverable and Reusable Catalyst
 Javad Safaei-Ghomi^{a*}, Reihaneh Masoomi^a

^aDepartment of Organic Chemistry, Faculty of Chemistry, University of Kashan, 51167 Kashan, I. R. Iran.

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Abstract

In this research an effective and appropriate method has been developed for one-pot synthesis of new N-phenyl fulleroisoxazoline using Fe₃O₄@SiO₂ nanoparticles as a green magnetically recyclable catalyst. The prepared catalytic samples were characterized by XRD, SEM, FT-IR and VSM. The products have been characterized by physical and spectroscopic data such as IR, ¹H NMR, ¹³C NMR, and MS analyses.

**Corresponding author:*

E-mail address:

safaei@kashanu.ac.ir

Phone: +98 31 5591 2385

Fax: +98 31 5552935

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1. Introduction

Fullerenes as the third allotrope of carbon were discovered by Kroto *et al.* [1] in 1985. The chemistry of fullerenes began to develop greatly after W. Kratschmer and D. Huffman [2] who found out a method for the preparation of fullerene soot by burning graphite rods in arc discharge in an inert atmosphere. Fullerene-based nanomaterials display an extensive range of interesting chemical, physical, biological [3-7] and other scientific properties [8-10]. Surface modification of fullerene is an attractive approach in materials science and pharmaceutical

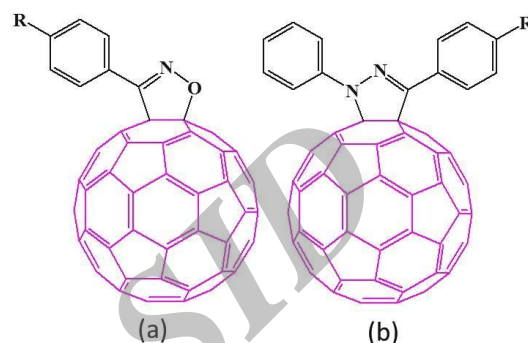
applications [5, 11, 12]. The electron withdrawing nature of the closed cage alkene C₆₀ makes it an excellent dienophile for cycloadditions [13-15], addition of organometallic reagents [16], and photo-induced electron transfer reactions [17]. Among the different types of cycloaddition reactions [18-21] available for the preparation of heterocyclic fullerene derivatives, 1,3-dipolar cycloadditions demonstrate a powerful tool due to the fact that C₆₀ behaves as an electron-deficient olefin. Synthesis of five-member cycles *via* the addition of 1,3-dipoles to alkenes is a typical organic reaction. In fact, 1,3-dipolar cycloaddition reactions are beneficial for the construction of

carbon-carbon bonds and for the preparation of heterocyclic compounds [22]. So far different 1,3-dipoles include azomethine ylides, diazo compounds, azides, nitrile oxides, nitrile ylides, nitrile imines, pyrazolinium ylides and carbonyl ylides have been reported to react with fullerenes [23-30]. Fullerene-based heterocyclic rings including fulleropyrrolidines [31], fullerindolines [32], fullerene-fused lactones [33], pyrazolo- and oxazolo- fullerenes [22] with noteworthy chemical, electrochemical, and photophysical properties can be readily synthesized through various methods such as addition of nitrile imine and nitrile oxide to C₆₀ [33-40].

Magnetic nanoparticles have received attractive attention because of their matchless physical and chemical properties and potential applications in different biomedical fields including targeted drug delivery, magnetic hyperthermia, magnetic resonance imaging (MRI), biosensing, and magnetic separation of biological materials [41-44]. In particular, Fe₃O₄ nanoparticles coated with a thin layer of silica have advantageous properties, such as stability and fixed catalytic activity [45]. Silica has been considered as a prominent material for encapsulating the magnetic nanoparticles due to its easy multi functionalization, good biocompatibility and excellent physicochemical stability [46, 47]. This effective shell is able to encapsulate the nanoparticles in an inert shell, which inherently improved their surface properties and prevents direct contact between the nanoparticles [48, 49].

In this context, we were prepared Fe₃O₄ nanoparticles by chemical co-precipitation method and subsequently coated with tetraethoxysilane (TEOS) *via* silanization reaction. In continuation of our researches on the application of various techniques for the synthesis of fulleropyrrazolines

and fulleroisoxazolines (Scheme 1) [50-52], we have successfully studied the catalytic synthesis of new *N*-phenyl fulleroisoxazoline derivative by 1,3-dipolar cycloaddition reaction *via* Fe₃O₄@SiO₂ core shell nanoparticles for the first time.



Scheme 1. The chemical structures of (a) fulleroisoxazoline and (b) fulleropyrazoline

2. Experimental

2.1. Materials and Apparatus

Crystalline C₆₀ powder used in this work was over 99.5% purity from Aldrich. All solvents and chemical reagents were purchased from Merck and Aldrich and used without further purification. Melting points were determined on Electro thermal 9200, and are not corrected. FT-IR spectrum was recorded on Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400-4000 cm⁻¹. ¹H NMR and ¹³C NMR spectra were recorded on Bruker Avance-400 MHz spectrometers in the presence of tetramethylsilane as internal standard. The mass spectra was performed by Finnigan-MAT-8430 mass spectrometer in m/z. Nanostructures were characterized using a Holland Philips Xpert X-ray powder diffraction (XRD) diffractometer (CuK, radiation, k = 0.154056 nm), at a scanning speed of 2 °/min from 10 ° to 100 ° (2θ). Scanning electron microscope (SEM) and energy dispersive

analysis X-ray (EDX) of nanoparticles was performed on a MIRA 3-TESCAN. Vibrating Sample Magnetometer (VSM) of nanoparticles was performed *via* Iran Nanotechnology Initiative Council (INIC).

2.2. Preparation of magnetic Fe₃O₄ nanoparticles

Typically, FeCl₃.6H₂O (2.7 g) and FeCl₂.4H₂O (1 g) were dissolved in 100 ml of 1.2 mmol/lit aqueous HCl by ultrasonic bath for 30 min. Then, 1.25 mmol/lit aqueous NaOH (150 ml) was added under vigorous stirring and a black precipitate was immediately formed. The resulting transparent solution was heated at 80 °C with rapid mechanical stirring under N₂ atmosphere. After stirring for 2 h, the precipitate was magnetically separated and washed thoroughly with water until the supernatant liquid reached neutrality (pH ~7). Finally, the black precipitates were washed twice by ethanol and dried in vacuum at 70 °C overnight to obtain Fe₃O₄ nanoparticles.

2.3. Preparation of nano-Fe₃O₄@SiO₂

Fe₃O₄ MNPs (1 g) were initially diluted *via* the sequential addition of water (20 ml), ethanol (60 ml), and concentrated aqueous ammonia (2 ml, 25 wt %). The resulting dispersion was then homogenized by ultrasonic. A solution of TEOS (0.5 ml) in ethanol (10 ml) was then added to the dispersion in a drop-wise manner under continuous mechanical stirring. After vigorous stirring for 16 h, the dark brown product were collected by an external magnetic. Then Fe₃O₄@SiO₂ MNPs was washed with ethanol three times and dried in a vacuum.

2.4. Preparation of N-phenylhydroxylamine

In a 500 ml beaker, equipped with a thermometer and mechanical stirrer, 7.18 g. of NH₄Cl was added to 250 ml. of distilled water and 13 ml of nitrobenzene. After vigorously stirring the mixture, 19.37 g. of zinc dust powder of 90% purity was added at about 15 minutes; the rate of addition should be such that the temperature rapidly rises to 60-65 °C and remains in this range until all the zinc has been added. After completion the reaction, the mixture was filtered at the pump to remove the zinc oxide, and washed with 100 ml of hot water. Then the filtrate was saturated with NaCl salt (about 60 g) and cooled in an ice bath for at least 1 hour to ensure maximum crystallization of the desired product. The pale yellow crystals of *N*-phenylhydroxylamine were obtained and pureed *via* crystallization from benzene–light petroleum ether or benzene.

N-Phenylhydroxylamine (2): Yellow needles crystals; Mp °C: 81; FT-IR (KBr) ν : 3300, 3129, 3051, 2966, 1600, 1590, 1521, 1420, 1378, 1306, 1059, 723, 616 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ (ppm): 6.40 (br., 2H, OH, NH), 6.99 (dd, 3H, ArH), 7.28 (t, 2H, ArH).

2.5. Synthesis of N-Phenyl- α -(4-nitrophenyl)nitron

A mixture of *N*-phenylhydroxylamine (4.96 g, 0.045 mol) and 4-nitrobenzaldehyde (6.84 g, 0.045 mol) in the minimum amount of ethanol was allowed to stand at room temperature overnight. The obtained precipitates were filtered and recrystallized from a mixture of 2:3 benzene:petroleum ether to afford the pure nitron (85%).

N-Phenyl- α -(4-nitrophenyl)nitron (4): Light yellow crystals; Mp 193 °C: FT-IR (KBr) ν : 3408,

3102, 2925, 1594, 1512, 1402, 1342, 1187, 1074, 858, 772, 688 cm^{-1} ; ^1H NMR (400 MHz, acetone- d_6) δ (ppm): 7.59 (t, 3H, ArH), 7.97 (d, 2H, ArH), 8.37 (d, 2H, ArH), 8.64 (t, 1H, ArH), 8.77 (d, 2H, ArH).

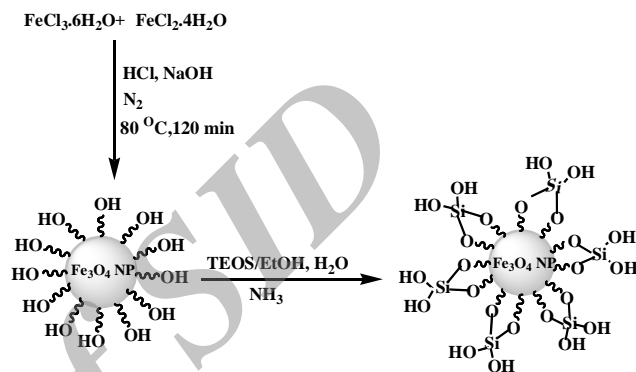
2.6. General procedure for the synthesis of *N*-(phenyl)- 3'- (4-nitrophenyl)- 3'-*H*-isoxazoline [4',5': 1,2] fullerene

A mixture of C_{60} (0.05 mmol, 36.0 mg), *N*-Phenyl- α -(4-nitrophenyl)nitron (0.05 mmol, 13.0 mg) and $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs (0.5 gr) were dissolved in 20 mL of toluene and stirred under nitrogen atmosphere under reflux conditions for a desired time. The course of the reaction was monitored by TLC with toluene as an eluent. After completion of the reaction, the reaction mixture was cooled and the catalyst was separated by an external magnet and reused for the next experiment. Then the solvent was evaporated in vacuum, and the residue was separated on a silica gel column using toluene to afford the desired cycloadduct.

N-(phenyl)- 3'- (4-nitrophenyl)- 3'-*H*-isoxazoline [4',5': 1,2] fullerene (6): Brown solid; Mp >300 $^\circ\text{C}$; FT-IR (KBr) ν : 3000, 2924, 1629, 1500, 1384, 1097, 797, 755, 695 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 - CS_2) δ (ppm) : 7.39 (dd, 1H, ArH), 7.53 (m, 5H, ArH), 8.17 (t, 2H, ArH), 8.32 (d, 1H, ArH); ^{13}C NMR (100 MHz, CDCl_3 - CS_2): δ (ppm) 85, 95, 109, 124 (2C), 125, 127, 130 (2C), 131 (2C), 134, 136, 138, 139, 140 (2C), 141, 142 (2C), 143 (2C), 144 (2C), 145 (2C), 146 (2C), 147, 148; 149, 151, 154, MS (EI, 70 eV): m/z (%) = 962 (M^+ , 10).

3. Results and discussion

Initially synthesis of highly stable Fe_3O_4 nanoparticles was occurred *via* iron(II) and iron(III) ions by co-precipitation technique [52]. For the surface modification, the Fe_3O_4 MNPs coated with a layer of silica using the TEOS reagent [54] to provide reaction sites for further functionalization (Scheme 2).



Scheme 2. Synthesis of Fe_3O_4 and silica coated Fe_3O_4 nanoparticles

The X-ray diffraction patterns of Fe_3O_4 and $\text{Fe}_3\text{O}_4@\text{SiO}_2$ are shown in Fig. 1. The position and respective intensities of all peaks emphasize well with standard XRD pattern of Fe_3O_4 representing retention of the crystalline cubic spinel structure during functionalization of MNPs. Characteristic peak of SiO_2 in core shell structure has been hidden under weak peak of Fe_3O_4 at $2\theta = 30$. The average Fe_3O_4 and $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs core diameter were calculated to be 5 and 6 nm from the XRD results by cherrer's equation, $D = k\lambda/\beta\cos\theta$ where k is a constant (generally considered as 0.94), λ is the wavelength of $\text{Cu K}\alpha$ (1.54 \AA), β is the corrected diffraction line full-width at half-maximum (FWHM), and θ is Bragg's angle [55].

The FT-IR spectra of magnetic nanoparticles are showed the band in the region of 571 cm^{-1} is related to the stretching vibrations of the Fe-O

bond (Fig. 2a) and the band at about 1050 cm^{-1} belongs to Si-O stretching vibrations (Fig. 2b).

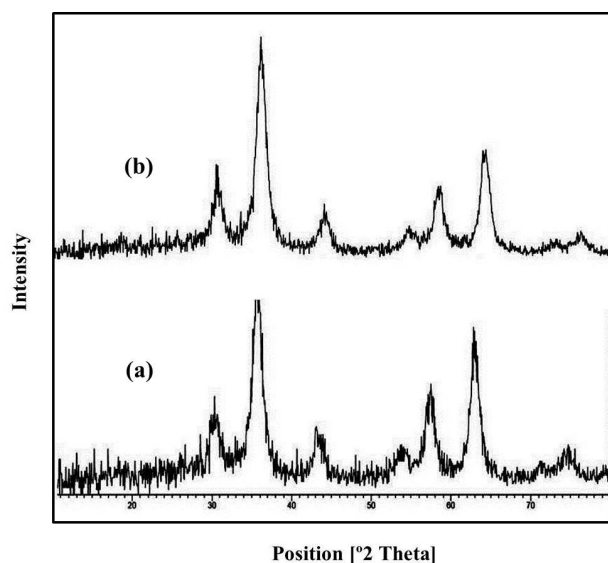


Fig. 1. XRD patterns of (a) Fe_3O_4 , (b) $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles

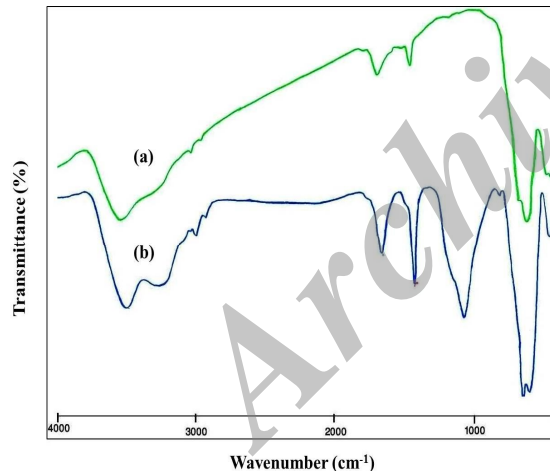


Fig. 2. The comparative FT-IR spectra of (a) Fe_3O_4 and (b) $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles

The SEM images of magnetic nanoparticles are shown in Fig. 3. These results show that MNPs were obtained with an average diameter of about 6-10 nm as confirmed by XRD analysis.

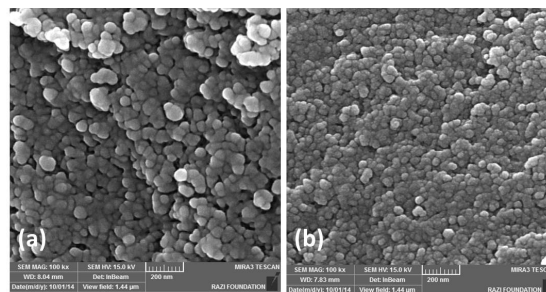


Fig. 3. The SEM images of (a) Fe_3O_4 and (b) $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles

The magnetization curve for Fe_3O_4 and $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles are shown in Fig. 3. Room temperature specific magnetization (M) versus applied magnetic field (H) curve measurements of the $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ indicate a saturation magnetization value (M_s) of 36.73 emu g^{-1} , lower than that of bare Fe_3O_4 MNPs (40.05 emu g^{-1}) due to the coated shell (Fig. 4).

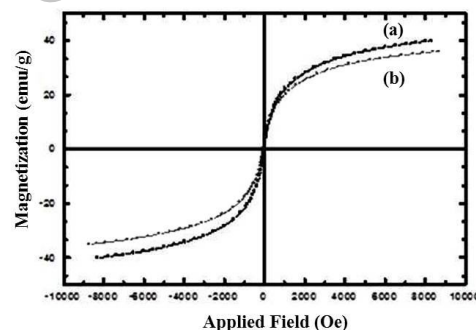


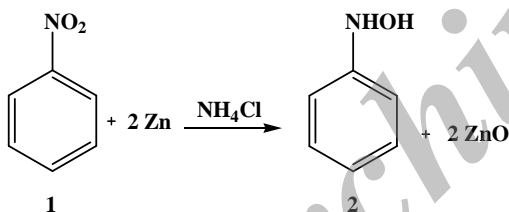
Fig. 4. The magnetization curves for the prepared (a) Fe_3O_4 and (b) $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles

Figure 5 shows the image of $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ microspheres that dispersed in ethanol. After a magnet was placed aside, the brown microspheres can be magnetized in 1 min, leaving a clear solution. That is to say, the $\text{Fe}_3\text{O}_4@ \text{SiO}_2$ nanoparticles were shown good magnetic responsibility even if the SiO_2 layer was increased to 6 nm.



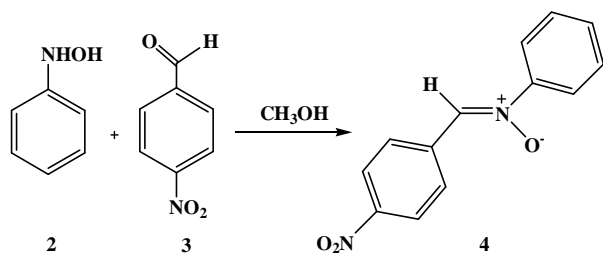
Fig. 5. Images of the ethanol solution (a) with dispersed magnetic $\text{Fe}_3\text{O}_4@\text{SiO}_2$ nanoparticles and (b) after applied magnetic field

Continuing research, novel synthesis of new *N*-phenylfulleroisoxazoline derivative through 1,3-dipolar cycloaddition reaction under reflux conditions is discussed. Phenylhydroxylamine was readily prepared by the reaction of nitrobenzene, Zn powder and ammonium chloride in water under thermal conditions [56]. After recrystallization from benzene–petroleum ether, it was purified enough to be used (Scheme 2).



Scheme 2. Synthesis of *N*-phenylhydroxylamine

In the next step, *N*-Phenyl- α -(4-nitrophenyl)nitron was synthesized in good yields according to the reported literature [57].



Scheme 3. Synthesis of *N*-Phenyl- α -(4-nitrophenyl)nitron

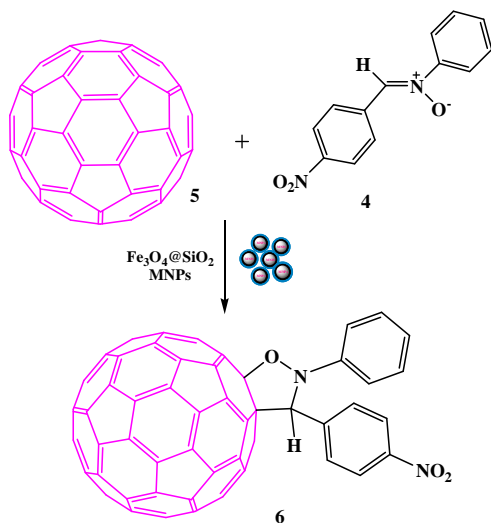
Herein, catalytic synthesis of new *N*-phenyl fulleroisoxazoline is studied as an efficient and complementary technique to promote 1, 3-dipolar cycloaddition reaction under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs. Cycloaddition reaction of C_{60} with nitron derivative under conventional (110°C) and in presence of nano catalyst of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs occurred to prepare the corresponding *N*-phenyl fulleroisoxazoline. To our satisfaction, when a mixture of C_{60} (0.05 mmol, 36.0 mg) and *N*-Phenyl- α -(4-nitrophenyl)nitron (0.05 mmol, 13.0 mg) was refluxed in 20 mL toluene for 48 h, any fullerene-based product was not prepared. Then for the examination of efficient and green influence of nanocatalyst in the respective procedure, the reaction was investigated under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs as novel magnetite nanocatalyst. It was found that with a suitable molar ratio, nano catalyst could influence on efficient synthesis of the fullerene adduct. At first various amount of catalyst were studied and the results were summarized in Table 1.

Table 1. The synthesis of (6) under a different amount of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs

No	Amount (g)	Yield (%)
1	0.1	NR ^a
2	0.2	NR
3	0.3	NR
4	0.4	22
5	0.5	35
6	0.6	36

^aNo Reaction

However cycloaddition reaction of C_{60} with *N*-Phenyl- α -(4-nitrophenyl)nitron in the presence of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs under reflux conditions occurred quickly (2-3 h), to prepare the corresponding *N*-phenyl fulleroisoxazoline in about 35% yield as described in Scheme 4.

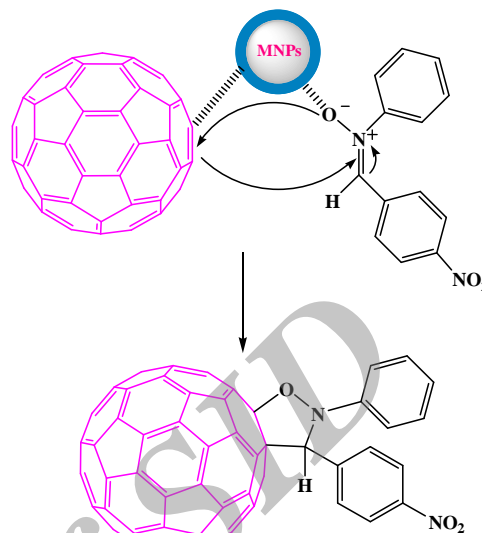


Scheme 4. Novel synthesis of *N*-(phenyl)-3'-(4-nitrophenyl)-3'-*H*-isoxazoline [4',5':1,2] fullerene

It was found that the procedure was a simple, mild and suitable method of constructing isoxazoline cycle on fullerene through 1,3-dipolar reaction and greatly accelerated under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs as novel magnetic nanocatalyst. In fact the experimental results showed that the reaction time could be shorted and the yield of the product increased under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs.

The plausible mechanism for the catalytic synthesis of *N*-(phenyl)-3'-(4-nitrophenyl)-3'-*H*-isoxazoline [4',5':1,2] fullerene is shown in Scheme 5. Initially, we assumed that $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs is an effective surface for orientation of nitron dipole versus C_{60} . In the presence of magnetic nanocatalyst, nitron and the double bond of fullerene is activated. Then, the subsequent 1,3-dipolar cycloaddition reaction occurred by this dipole with fullerene under the efficacious surface of nanocatalyst. In this mechanism the $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs perform as Lewis solid acid and activate the $\text{C}=\text{C}$ bond of C_{60} for proper reaction with reactive nitron. In fact cycloaddition reaction could be catalyzed

using $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs through a suitable contact between the substrates.



Scheme 5. Plausible mechanism for the synthesis of *N*-(phenyl)-3'-(4-nitrophenyl)-3'-*H*-isoxazoline [4',5':1,2] fullerene under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs

The corresponding product was obtained in good yields and reaction time under $\text{Fe}_3\text{O}_4@\text{SiO}_2$ MNPs. The new C_{60} -based cycloadduct was fully characterized by their FT-IR, ^1H NMR, ^{13}C NMR and MS analyses. In the mass spectrum (EI, 70 eV), the peak at m/z 960 is related to molecular ion of M^+ . In the IR spectra, the stretching frequency of aromatic $\text{C}=\text{C}$ is produced in the area between $1480\text{--}1600\text{ cm}^{-1}$. Also stretching frequency of $\text{C}-\text{N}$ of isoxazoline ring appears at 1629 cm^{-1} . In the ^1H NMR spectra, the appearance of $\text{CH}-\text{N}$ proton signal in *N*-Phenyl- α -(4-nitrophenyl)nitron (4) and the related fulleroisoxazoline spectra appear around the $\delta = 7.2\text{--}7.5\text{ ppm}$. The other signals about $\delta = 7.2\text{--}8.3\text{ ppm}$ are assigned by protons of $\text{CH}-\text{CH}$ of aromatic rings in nitron and *N*-phenyl fulleroisoxazoline (Fig. 6).

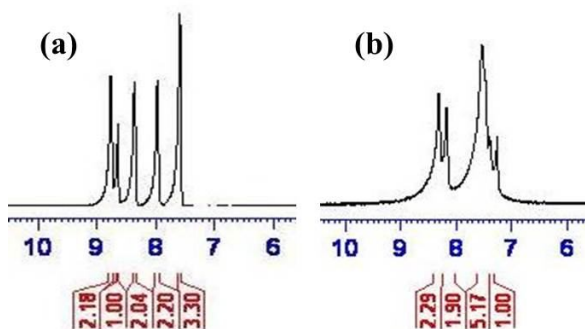


Fig. 6. ^1H NMR spectrum of (a) *N*-Phenyl- α -(4-nitrophenyl)nitrene; (b) *N*-(phenyl)-3'-(4-nitrophenyl)-3'-*H*-isoxazoline [4',5': 1,2] fullerene

4. Conclusion

In conclusion, we have developed an appropriate and highly efficient protocol for the synthesis of *N*-(phenyl)-3'-(4-nitrophenyl)-3'-*H*-isoxazoline [4',5': 1,2] fullerene using $\text{Fe}_3\text{O}_4@\text{SiO}_2$ nanoparticles under reflux conditions. This procedure has several obvious advantages including avoiding the use of harmful catalyst, cost-effective catalyst, recyclability and reusability of the magnetic catalyst, convenient workup of the reaction, good yield, short reaction time and simplicity of the methodology. We expect this approach will disclose extensive purposes in the field of fullerene chemistry, green synthesis, promising candidates for materials science and biological applications and modeling of novel nanocatalysts in the future.

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