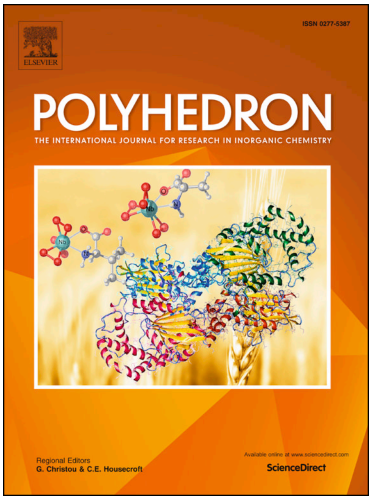

Neodymium immobilized on Fe_3O_4: A new and recoverable catalyst for oxidation reactions and synthesis of 5-substituted 1H–tetrazoles in green condition

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Neodymium immobilized on Fe_3O_4: A new and recoverable catalyst for oxidation reactions and synthesis of 5-substituted 1H–tetrazoles in green condition

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Abstract

Magnetic nanoparticle functionalized with neodymium complex as catalyst and characterized by SEM, XRD, FT-IR, TGA, EDX and ICP techniques. Then, catalytic activity of prepared catalyst was examined in the synthesis of tetrezoles and oxidation reactions in which observed short reaction time and high yield in these reactions.

Keywords Fe_3O_4, Nd, Green, 5-substituted tetrazoles, Sulfoxides, Disulfides

Introduction

According to a green chemistry, one of the most important principles of chemistry, the use of heterogeneous catalysts is nowadays a well-established method for control of pollution. Thus, heterogeneous catalysts have extensively developed in organic synthesis compared to homogenous catalysts their because of economic and industrial aspects [1-4]. In order to preparing heterogeneous catalysts, various supports have been proposed [5-8]. In this regard, magnetic nanoparticles are one of the important supports due to facilitate the access of substrates to active sites, easy access to surface in order to support functionalized, high dispersion, excellent stability (chemical and thermal), easy synthesis and easy separation [9-18]. In the last few years, superparamagnetic Fe_3O_4 nanoparticles modified with several metal complexes such as Ni, Cu, Pd, Co, VO, Zn, Zr, Cd, Mn and Cr in order to synthesis of heterogeneous catalysts for organic synthesis such as oxidation reactions and synthesis of 5-substituted tetrazoles [19-24]. On the other hand, 5-substituted tetrazoles, sulfides and disulfides as versatile organic reagent are important and necessary component in biological processes, chemical and pharmaceutical industry. The product of oxidation of sulfides and thiols have received considerable importance in the development of sensors, oil-sweetening processes and vulcanizing agents, etc [25-27]. Tetrazole compounds have been widely used for more than one hundred because they are nitrogen-rich heterocycles with a wide range of applications such as catalysis technology, ligands in coordination chemistry, heterocyclic chemistry, effective stabilizers of metallopeptide structures in organometallic chemistry, stable surrogates for carboxylic acids [28-32].

Herein, we interest to report of the synthesis and characterization of Nd complex supported on Fe_3O_4 as a thermostable catalyst for synthesis of 5-substituted tetrazoles, disulfides and sulfoxides in neat condition. It was also indicated that up to date, the reports was not from Nd complex supported on Fe_3O_4 in synthesis above-mentioned compounds neat condition.

Experimental

Materials and physical measurements

All solvents and materials for performance of this project are purchased from Merck and Sigma-Aldrich and used without further purification. The elemental analysis and exact amount of Nd were determined by Energy-dispersive X-ray spectroscopy (EDAX, TSCAN) and inductively coupled plasma-optical emission spectrometry (ICP-OES), respectively. The surface morphology of nanostructures and texture properties were determined by SEM using FESEM-TESCAN MIRA3 and Powder X-ray diffraction (XRD) measurements using Co radiation source with a wave length = 1.78897 Å, 40 kV, respectively. Melting points of all products were determined with an Electrothermal 9100 apparatus. Thermal stability of the nanocatalyst and fourier transform infrared (FT-IR) spectra of all samples performed by TGA measurement Shimadzu DTG-60 instrument and a Bruker VERTEX 80 v, respectively.

Preparation of Fe_3O_4@Nd nanocatalyst

Following the method reported in the literature, we obtained Fe_3O_4 nanoparticles according to a previous report by Darabi et al [22]. Then, the modification of Fe_3O_4 was performance using the reflux of mixture contain 1 g of MNPs powder and 1.5 g of tryptophan ligand in distilled water (30 mL) for 24 h. After the reflux of mentioned mixture, the final prtoduct was separated by an external magnet, and washed with ethanol/water and dried to give functionlized Fe_3O_4 by tryptophan. Then, Fe_3O_4@Nd nanocatalyst was obtained by mixing 1 g of functionlized Fe_3O_4 by tryptophan and 1.09 g Nd $(NO_3)_3.6H_2O$ in 30 mL absolute ethanol at reflux condition for 16 h.

General procedure for the oxidation reactions

To a stirred suspension of Fe_3O_4@Nd in H_2O at

mmol) was added under solvent-free condition. After the completion of reaction (monitored by TLC) in appropriate time, the separated catalyst (using by magnetic) washed with ethyl acetate to obtained pure sulfoxide or disulfide derivatives.

General procedure for the synthesis of 5-substituted tetrazoles

To a stirred suspension of Fe_3O_4@Nd (5 mg) in H_2O (2 mL) at 100 °C, nitrile (1 mmol) and sodium azide (1.2 mmol) added to reaction mixture in appropriate time. After the completion of reaction (monitored by TLC), the catalyst separated by magnetic and then mixture was diluted with HCl (4 N, 10 mL). Finally, the resultant organic layer extracted and washed with ethyl acetate to obtain corresponding tetrazoles.

Selected spectral data

Methyl phenyl sulfoxide ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ_H= 2.65 (s, 3H), 7.54–7.69 (m, 2H), 7.61–7.64 (m, 3H).

5-(4-Nitrophenyl)-1H-tetrazole ^{1}H NMR (400 MHz, DMSO, ppm): δ_H= 8.28–8.31 (m, 2H), 8.39–8.43 (m, 2H).

5-(3-Nitrophenyl)-1H-tetrazole ^{1}H NMR (400 MHz, DMSO, ppm): δ_H= 7.93 (t, *J*= 8.4, 2H), 8.43–8.50 (m, 2H), 8.51–8.52 (s, 1H).

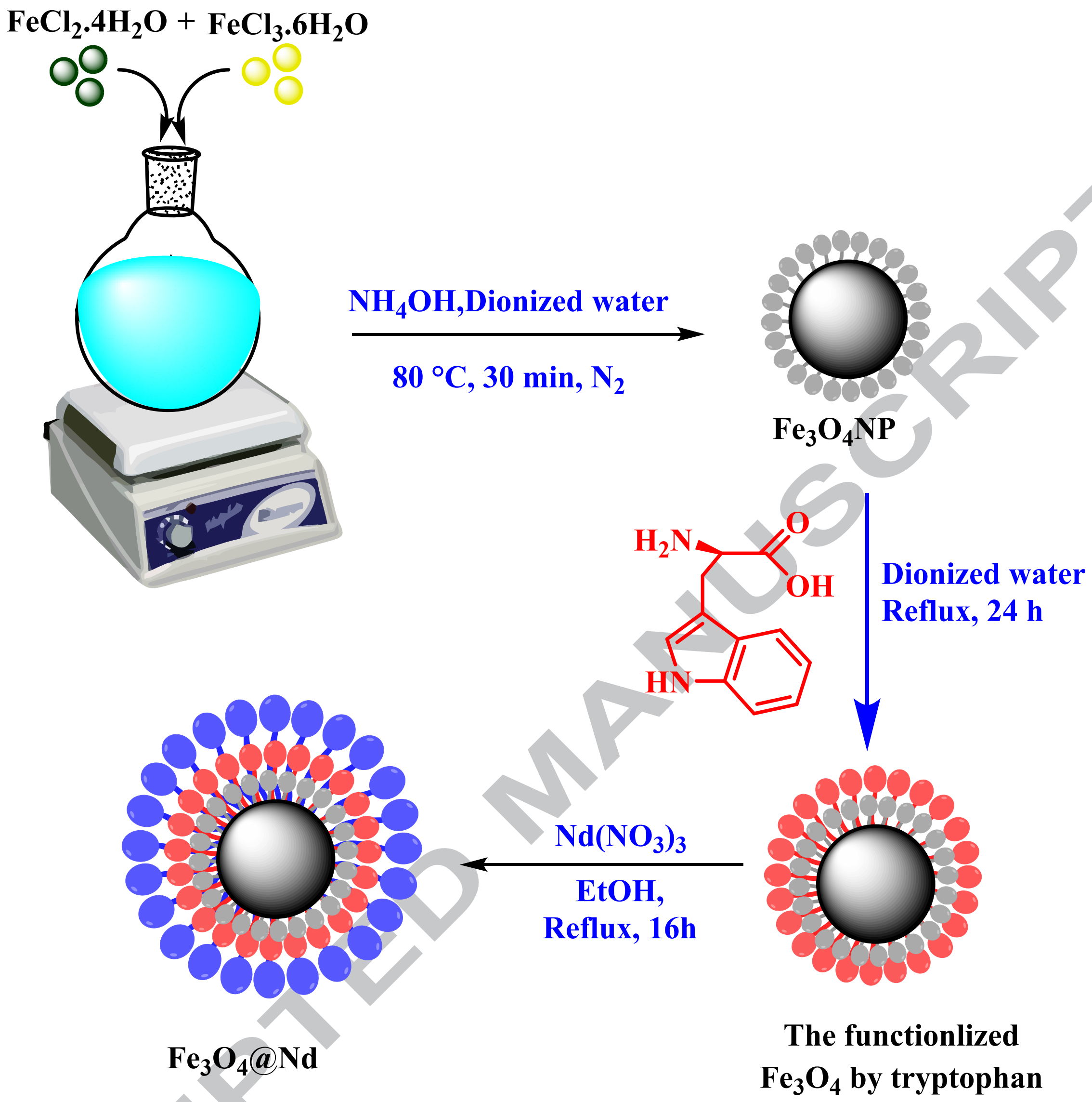
5-(4-Chorophenyl)-1H-tetrazole ^{1}H NMR (400 MHz, DMSO, ppm): δ_H= 7.85–8.1 (m, 4H).

1,2-Di(naphthalen-2-yl)disulfane ^{1}H NMR (400 MHz, DMSO): δ_H= 7.50–7.54 (m, 4H), 7.66–7.77 (m, 2H), 7.92–7.96 (m, 4H), 7.98-8.03 (m, 2H), 8.05 (s, 2H) ppm.

Results and discussion

Catalyst characterization

After successful synthesis of Fe_3O_4@Nd, SEM, FT-IR, EDX, XRD, TGA, ICP and VSM techniques were used to obtain the structure of nanocatalyst.



Scheme 1. Synthesis of $Fe_3O_4@Nd$.

In order to successful synthesis of catalyst, FT-IR spectra of Fe_3O_4, functionlized Fe_3O_4 by tryptophan and $Fe_3O_4@Nd$ nanoparticles was recorded in the 400–4000 cm^{-1}. As shown in Fig. 1, the O-H and Fe-O stretching vibration at approximately 3400-3500 and 580 cm^{-1}, respectively, in the FT-IR spectra of Fe_3O_4, functionlized Fe_3O_4 by tryptophan and $Fe_3O_4@Nd$ was confirmed the presence of Fe_3O_4 at all samples. In the spectrum of functionlized Fe_3O_4 by tryptophan, the characteristic absorption bands around 2850-2950 and 1620 cm^{-1} is correspounded of the aliphatic C-H and C=N stretching vibration, respectively.

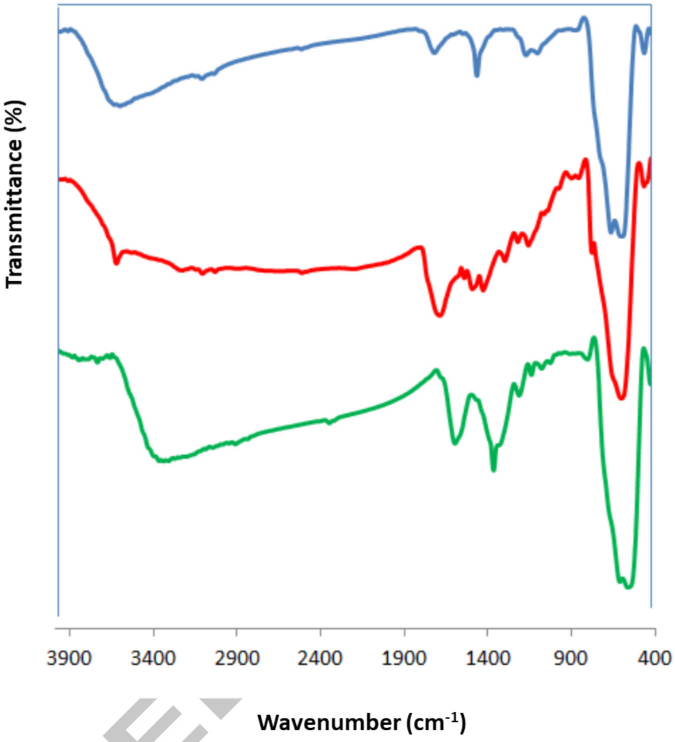


Fig. 1. FT-IR spectrum of Fe_3O_4 (blue), functionlized Fe_3O_4 by tryptophan (red), Fe_3O_4@Nd catalyst (green).

Thermo-gravimetric analysis (TGA) of the organic groups supported on the surface of bare Fe_3O_4 nanostructure is indicated in Fig. 2. In TGA curve of catalyst, the first step of weight loss below 100 °C corresponds to the removal of the physically and chemically adsorbed solvents or surface hydroxyl groups, and the other weight loss in the range of 100-600 °C (9%) corresponds to the decomposition of the organic layers on the surface of Fe_3O_4 in which verified the well grafting of organic groups on the magnetic nanopartcles.

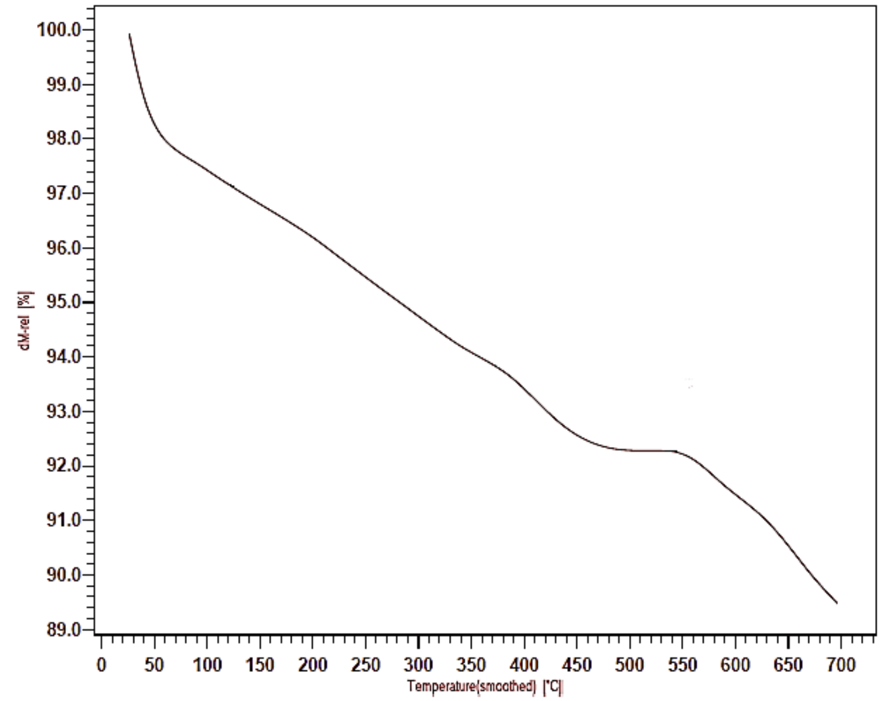


Fig. 2. The TGA diagram of Fe_3O_4@Nd.

SEM images of the Fe_3O_4@Nd in Fig. 3 at different magnifications were indicated that the mentioned nanocatalyst was made up of uniform spherical particles with size approximately 34 nm. It should be noted that the obtained morphology assisted to maximize the simultaneous increasing of the catalyst efficiency and frequency of molecular interactions or decreasing activation energy of rate determining step.

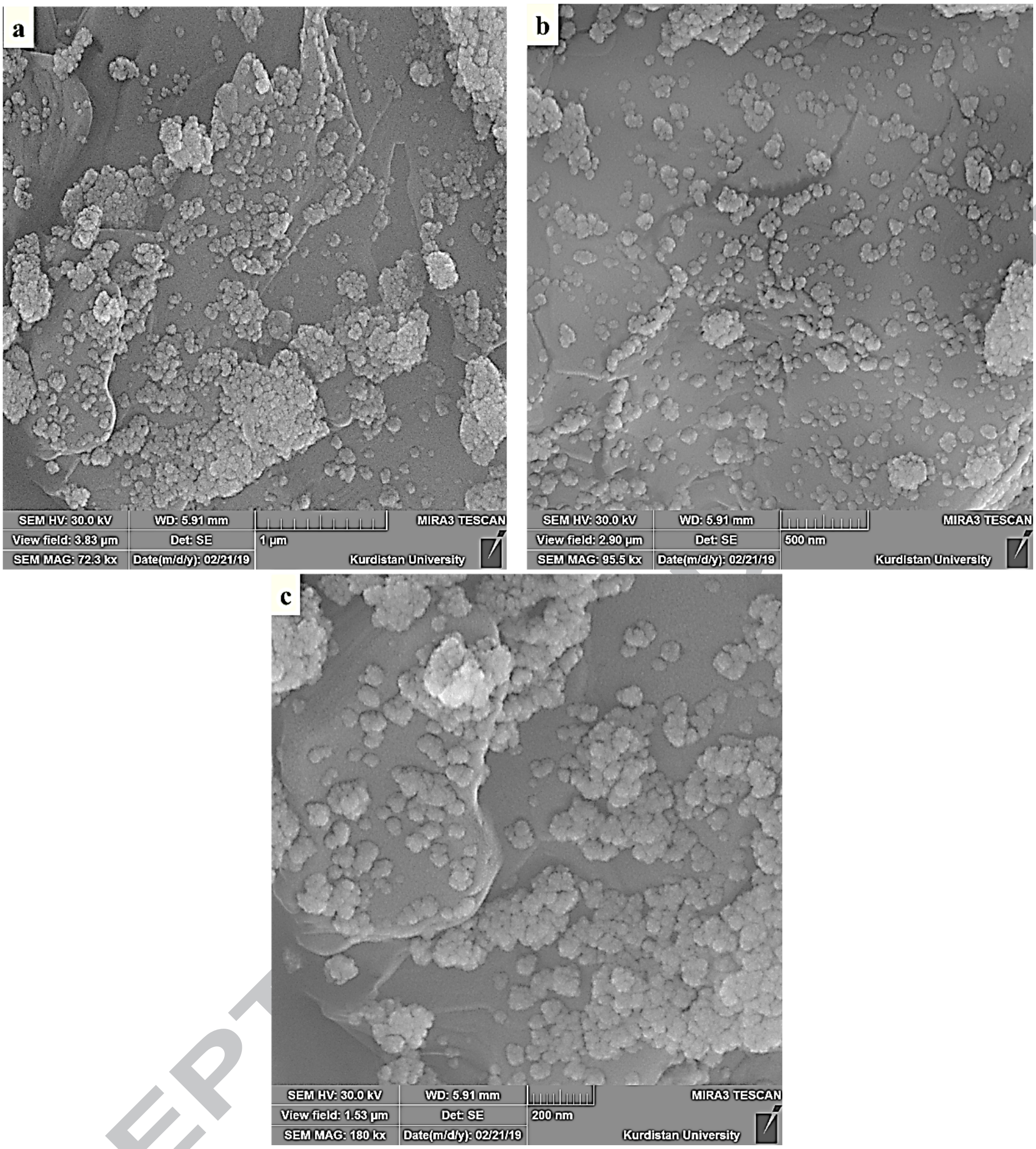


Fig. 3. SEM images of Fe_3O_4@Nd catalyst at 1µ (a), 500 nm (b) and 200 nm (c) magnifications**.**

The energy-dispersive X-ray spectroscopy (EDX) analysis of Fe_3O_4@Nd nanocatalyst was confirmed the presence of Fe, O, N, C and Nd specie in the obtained catalyst (Fig. 4), and amount of Nd loaded on modified magnetic nanoparticles was obtained 0.22 mmol g^{-1}.

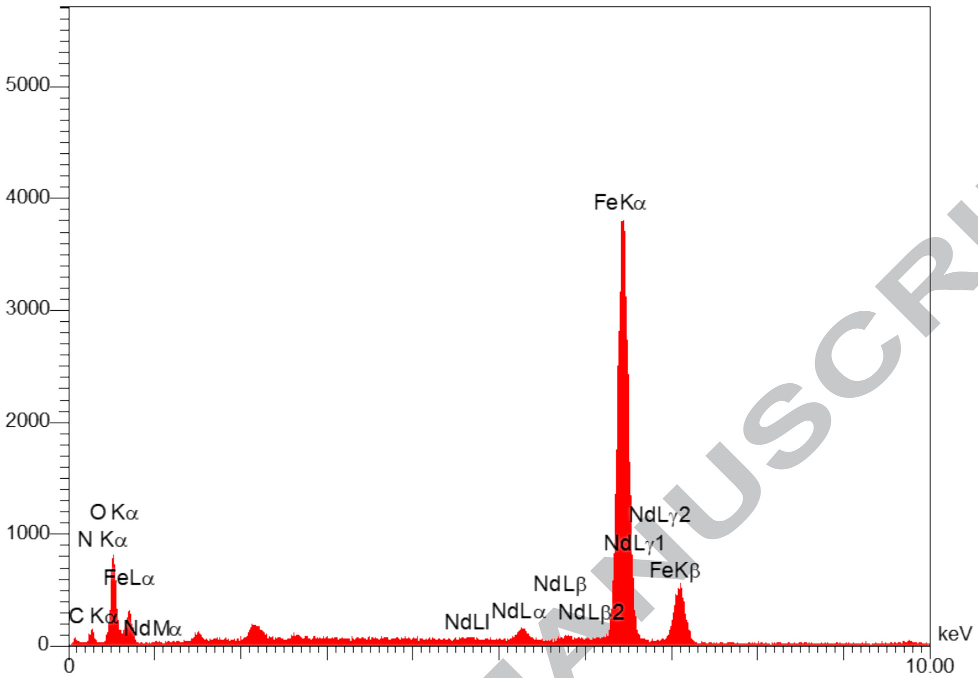


Fig. 4. EDX spectrum of Fe_3O_4@Nd.

The structural and textural properties of the Fe_3O_4@Nd nanocatalyst was determined by powder X-ray diffraction. As seen in Fig.5, the presence of a six peaks assigned to (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) reflections was verified the magnetite phase (JCPDS#01-075-0449) of Fe_3O_4 which crystallized in the cubic system. Also, the XRD pattern of prepared nanocatalyst indicated that the Fe_3O_4 phase has not been changed during the modifications [22].

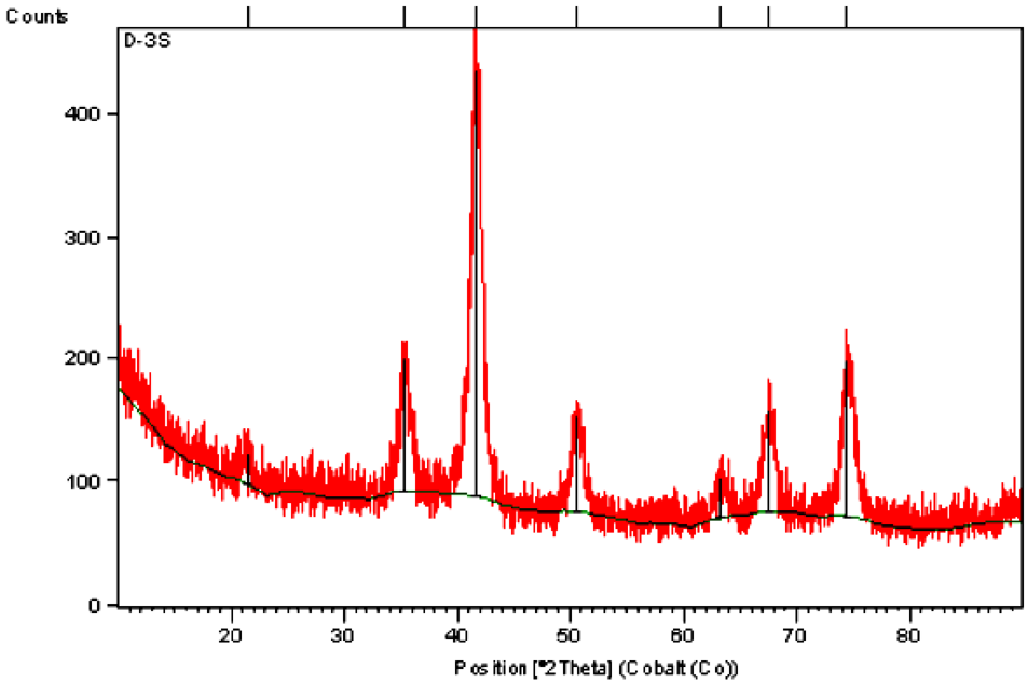
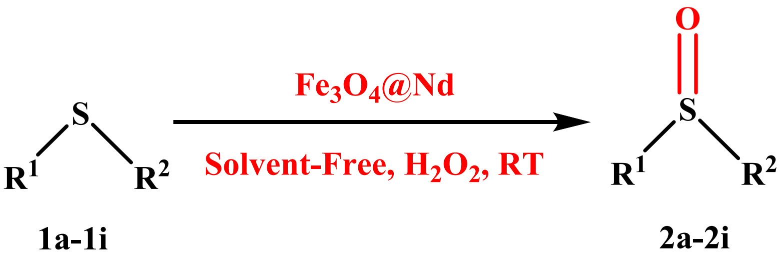


Fig.5. XRD pattern of Fe_3O_4@Nd.

Catalytic studies

Also, we tested catalytic activity of Fe_3O_4@Nd in the oxidation reactions (Scheme 2 and 3) and 5-substituted tetrazoles (Scheme 4). After the design of various experiments in order to optimize of the reaction conditions such as solvent, amount of catalyst and H_2O_2, the best results were obsvered in the presence of 5 mg Fe_3O_4@Nd and 0.5 mL H_2O_2 under solvent free (Table 1).



Scheme 2. Fe_3O_4@Nd catalyzed the oxidation of sulfides to sulfoxide.

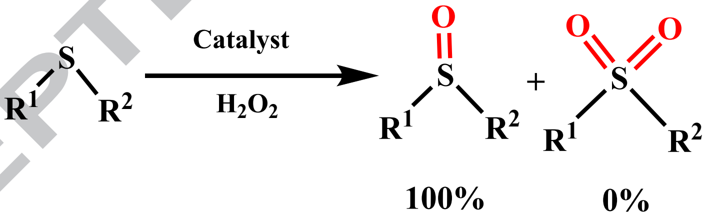
Table 1. Optimization of oxidation of sulfides to the corresponding sulfoxides using Fe_3O_4@Nd nanoparticles under various conditions.

Entry	Solvent	H_2O_2	Catalyst (mg)	Time (min)	Yield (%)[a]
1	Solvent-Free	0.5	3	45	65
2	Solvent-Free	0.5	5	10	96
3	Solvent-Free	0.5	7	10	99
4	Solvent-Free	0.4	7	15	92
5	Ethanol	0.5	7	55	81
6	Ethyl acetate	0.5	7	60	60
7	Acetonitrile	0.5	7	80	55

[a] Isolated yields.

Then, Fe_3O_4@Nd catalytic activity investigated in the wide range of sulfides with different functional groups in which observed results good to excellent (Table 2).

More importantly, because of mild conditions of described heterogeneous systems, there is no overoxidation to sulfone for oxidation of sulfides.



Scheme 3. Oxidation of sulfides to the corresponding sulfoxides.

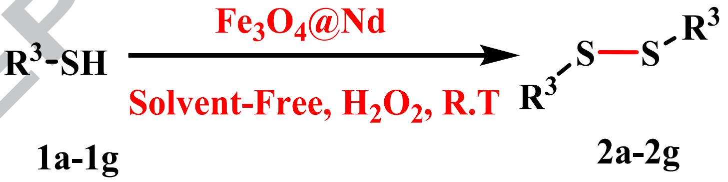
Table 2. Oxidation of sulfides to the sulfoxides in the presence of Fe_3O_4@Nd.

Entry	Substrate	Product	Time (min)	Yield (%)[a]	TON	TOF (h^{-1})	M.p. (°C)
1	Dipropylsulfide	2a	22	94	854	2308	Oil [11]
2	Diethylsulfide	2b	25	95	864	2057	Oil [11]
3	Dibenzylsulfide	2c	17	88	800	2857	127 [19]

4	Benzylphenylsulfide	2d	35	95	864	1490	117-120 [11]
5	Tetrahydrothiophene	2e	5	93	845	10181	Oil [21]
6	Ethylphenylsulfide	2f	12	92	836	4180	Oil [21]
7	Diphenylsulfide	2g	25	94	854	2033	61 [21]
8	3,3′-Thiodipropionic acid	2h	20	88	800	2424	206-208 [21]
9	Methylphenylsulfide	2i	10	96	873	5456	Oil [21]

[a] Isolated yields

Also, the synthesis of disulfide derivatives was investigated in the presence of Fe_3O_4@Nd catalyst (Scheme 4). Initially, condition of oxidation of 4-methylthiophenol was optimized that the best results were obtained in the presence of 5 mg of catalyst and 0.4 mL H_2O_2 under solvent free (Table 3). Melting points of the obtained products in Table 3 were 40-44 °C.



Scheme 4. Fe_3O_4@Nd catalyzed the oxidation of thiols to disulfides.

Table 3. Optimization of oxidative coupling of thiols into disulfides using Fe_3O_4@Nd under various conditions.

Entry	Solvent	H_2O_2	Catalyst (mg)	Time (min)	Yield (%)[a]
1	Solvent-Free	0.5	3	40	55
2	Solvent-Free	0.5	7	17	98
3	Solvent-Free	0.5	5	20	94
4	Solvent-Free	0.4	5	22	94
5	EtOH	0.5	5	50	80

6	Ethyl acetate	0.5	5	85	88
7	Acetonitrile	0.5	5	80	81

[a]Isolated yields.

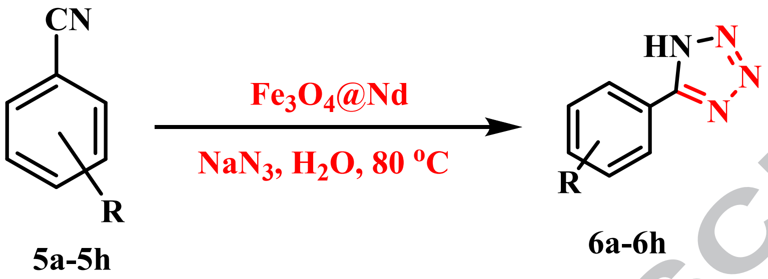
Then, in order to expand our study on synthesis of disulfides, a wide range of thiols was oxidated under the optimized condition in which indicated that products obtained in short time and good to excellent yiels (Table 4).

Table 4. Oxidative coupling of thiols into disulfides using H_2O_2 in the presence of Fe_3O_4@Nd.

Entry	Substrate	Product	Time (min)	Yield (%)[a]	TON	TOF (h^{-1})	M.p. (°C)
1	4-Methylbenzenethiol	4a	22	94	854	2308	41-43 [21]
2	Naphthalene-2-thiol	4b	20	94	854	2588	133 [11]
3	2-Mercaptobenzoic acid	4c	80	92	836	629	273 [11]
4	Benzo[d]thiazole-2-thiol	4d	40	96	873	1323	173-176 [21]
5	2-Mercaptoethanol	4e	35	88	800	1379	Oil [21]
6	Benzo[d]oxazole-2-thiol	4f	35	97	882	1521	92 [11]
7	Benzyl mercaptan	4g	25	98	891	2121	57 [21]

[a] Isolated yields

Finally, the synthesis of 5-(3-nitrophenyl)-1H-tetrazole was investigated in the different conditions such as solvent, temperature and amount of catalyst (Scheme 5) in which observed that the best results were obtained with 4 mg of Fe_3O_4@Nd and H_2O as green solvent 80 °C (Table 5).



Scheme 5. Fe_3O_4@Nd catalyzed the one-pot synthesis of 5-substituted tetrazoles.

Table 5 Optimization of reaction conditions for synthesis of 5-(3-nitrophenyl)-1H-tetrazole in the presence of Fe_3O_4@Nd.

Entry	Solvent	Temp (°C)	Catalyst (mg)	Time (min)	Yield (%)[a]
1	PEG	80	6	15	90
	EtOH	80	6	25	92
2	DMSO	80	6	20	90
4	H_2O	80	6	15	92
5	H_2O	80	8	10	95
6	H_2O	80	4	8	92
7	H_2O	80	2	35	75
8	H_2O	60	4	20	80
9	H_2O	100	4	8	94

[a] Isolated yields.

After optimization of reaction condition, a scope of the reaction, a series of aromatic tetrazoles was synthesized at same condition and results of this study were summarized in Table 6.

Table 6 Synthesis of 5-substituted 1H-tetrazole derivatives in the presence of Fe_3O_4@Nd under optimized reaction conditions.

Entry	Substrate	Product	Time (min)	Yield (%)^a	TON	TOF (h^{-1})	M.p. (°C)
1	Phthalonitrile	4a	25	90	1000	2380	202-205 [21]
3	3-Nitrobenzonitrile	4b	8	92	1022	7861	156-158 [11]
4	2-Chlorobenzonitrile	4c	55	95	1055	1152	182 [11]
5	2-Hydroxybenzonitrile	4d	30	91	1011	2022	221-223[11]
6	4-Hydroxybenzonitrile	4e	15	94	1044	4176	236-238 [21]
7	4-Chorobenzonitrile	4f	55	88	978	1068	257 [21]
8	4-Nitrobenzonitrile	4g	10	97	1078	6737	223 [11]
9	Benzonitrile	4h	80	88	978	735	21-212 [21]

^{a}Isolated yields.

Consideration the reusability of catalyst is a one of important factors in dustry, the reusability of the oxidation of methyl phenyl sulfide, coupling of 4-methylthiophenol and 5-(4-nitrophenyl)-1H-tetrazole was investigated. (Fig. 6).

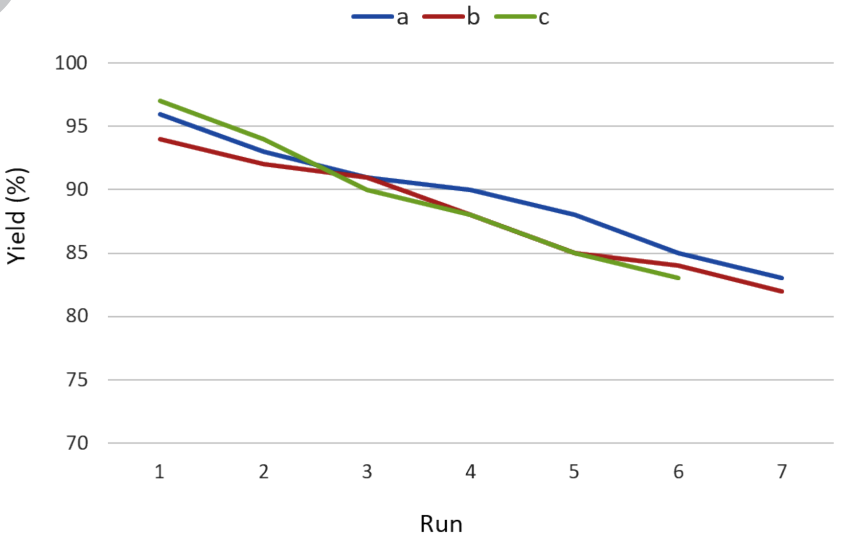


Fig. 6. Reusability of Fe_3O_4@Nd in the oxidation of methyl phenyl sulfide (a), coupling of 4-methylthiophenol (b) and 5-(4-nitrophenyl)-1H-tetrazole (c).

The nature of the recovered catalyst was investigated by EDX (Fig. 7) and FT-IR (Fig. 8) analysis. It was be observed that the catalyst can be recycled without any significant change in its structure.

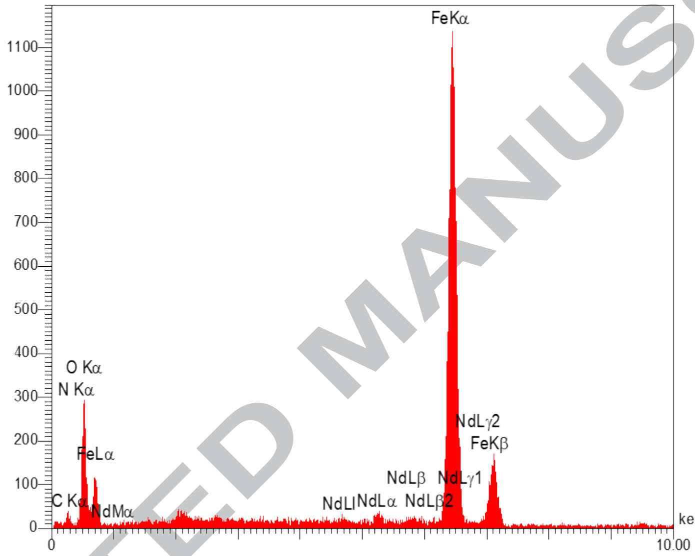


Fig. 7. EDX spectrum of recovered Fe_3O_4@Nd catalyst.

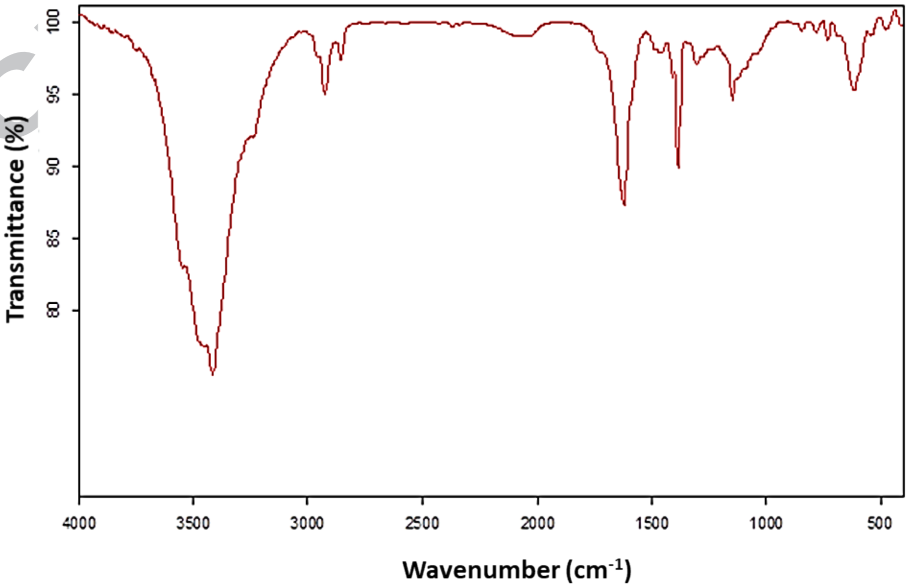


Fig. 8. FT-IR spectrum of recovered Fe_3O_4@Nd catalyst.

Comparison of the catalyst

Also, the obtained results of the oxidation of methylphenyl sulfide in this methodology were shown ease of operation, short reaction time, commercially available materials and high yield in the compare with previously reported methods (Table 7).

Table 7. Comparison of Fe_3O_4@Nd for the oxidation of methylphenyl sulfide with previously reported procedure.

Entry	Substrate	Catalyst	Time (min)	Yield (%)[a]
1	Methylphenylsulfide	Silica sulfuric acid/H_2O_2	60	72 [33]
2	Methylphenylsulfide	Fe_3O_4-Adenine-Ni	55	98 [34]
3	Methylphenylsulfide	Cu(II)–Schiff base complex- Fe_3O_4/H_2O_2	45	90 [35]
4	Methylphenylsulfide	VO-2A3HP-MCM-41	120	96 [36]
5	Methylphenylsulfide	$2NaBO_3.4H_2O$(I)/KBr	120	57 [37]
6	Methylphenylsulfide	Zr-oxide@MCM-41	300	98 [38]
7	Methylphenylsulfide	VO 2F$(dmpz)_2$	300	95 [39]
8	Methylphenylsulfide	Fe_3O_4	10	Trace
9	Methylphenylsulfide	Fe_3O_4@Tryptophan	10	Trace
10	Methylphenylsulfide	Fe_3O_4@Nd	10	96 [this work]

[a]Isolated yields.

Conclusion

In summary, in order to synthesis of Nd magnetic catalyst, Nd complex was successfull immobilized on Fe_3O_4. Then this nanocatalyst characterized by versatile chemical and physical techniques such as SEM, XRD, FT-IR, TGA, EDX and ICP and its catalytic behavier studied in the synthesis of 5-substituted tetrazoles and the oxidation reactions in green condition in which observed outstanding properties from present procedure such as ease of separation via magnetic field, high yield, the use of Nd as a new active site in organic reaction, high reusability of the catalyst.

Acknowledgements

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Graphical Abstract

Neodymium immobilized on Fe_3O_4 as recoverable catalyst

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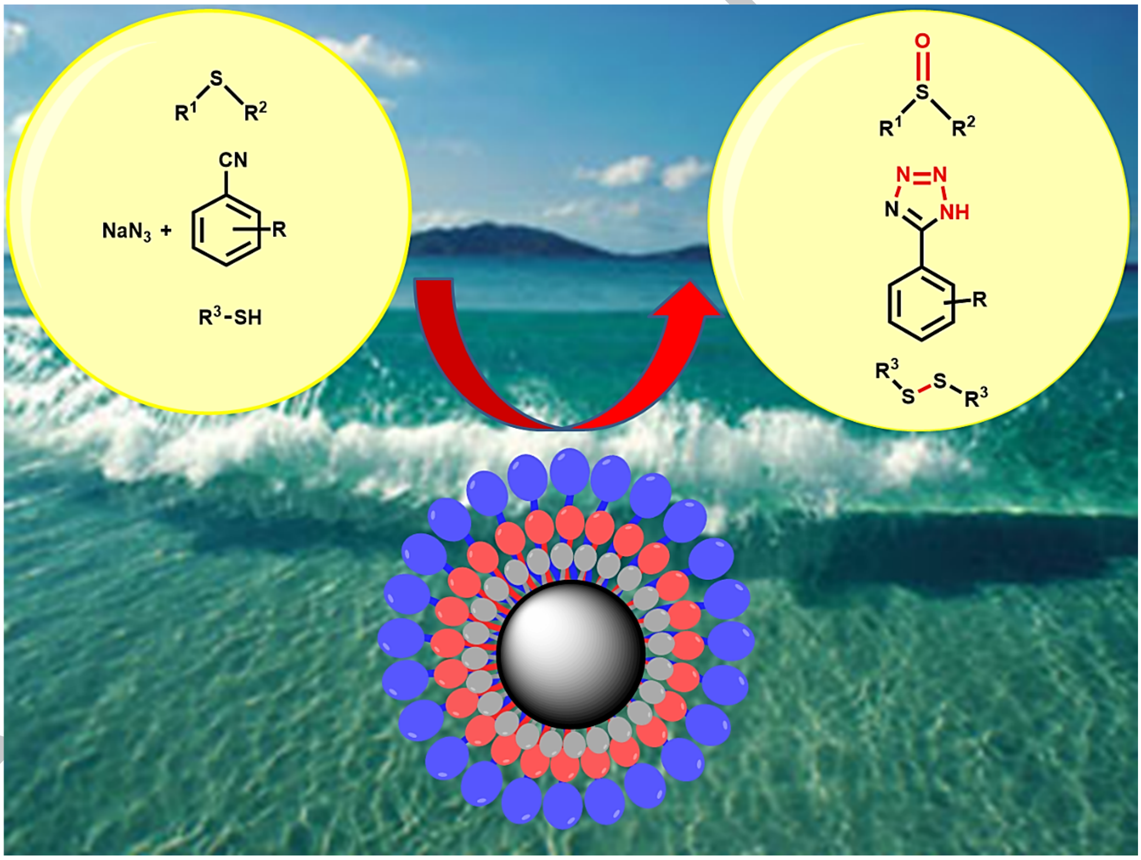

Graphical Abstract - Synopsis

The present work, we prepared a novel catalyst by linking Nd complex on Fe_3O_4 nanoparticles as green catalyst and characterized by FT-IR, SEM, EDX, ICP-OES, XRD and TGA methods. Then, catalytic activity of this catalyst was tested in the oxidation reactions and synthesis of tetrazoles. Easy preparation and easy separation, chemical and hydrothermal stability, the use of inexpensive materials, high reusable are outstanding properties of this protocol.