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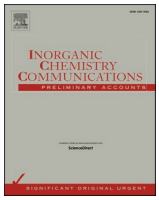

Research Article

Fabrication of a recyclable magnetic palladium nanocatalyst for generation of 5-phenyl-1*H*-tetrazoles from aromatic aldehydes

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ABSTRACT

A potent magnetic nanocomposite, Pd NPs/Fe_3O_4, has been developed by deposition of palladium nanoparticles over magnetic iron oxide nanoparticles using an extract from *Mentha piperita* leaves in an aqueous setting. The properties of the produced catalyst were investigated by using varies physicochemical methods, such as TEM, FE-SEM, XRD, EDX, VSM, ICP-OES, and elemental mapping. A novel and effective direct strategy for producing 5-Phenyl-1*H*-tetrazoles with good yields has been formulated by apply the fabricated Pd NPs/Fe_3O_4 catalyst. The synthesis process incorporated a three-component and one-pot [3 + 2] cycloaddition condensation that included sodium azide, hydroxylamine, and aldehyde, all catalyzed by Pd NPs/Fe_3O_4. The proposed mechanism likely consists of the in situ generation of nitrile as intermediate, which then undergo a [3 + 2] cycloaddition with NaN_3. The catalyst demonstrated the capability of being magnetically recovered and reused for a minimum of 6 cycles without a significant drop in catalytic efficiency. This approach consists additional benefits for the production of 5-Phenyl-1*H*-tetrazoles, such as easy access to starting substrates, simple reaction system, straightforward execution, and perfect yields.

1. Introduction

5-Phenyl-1*H*-tetrazoles shown a valuable class of polyaza-heterocyclic material that have been thoroughly researched due to their diverse applications. These compounds demonstrate a variety of potential biological effects, including anti-allergic [1], antibiotic [2], antagonist [3], antiviral properties [4], and antihypertensive [5]. Furthermore, tetrazoles play a crucial role in the formulation of numerous contemporary pharmaceuticals [6]. Recently, they have been employed to connect arylthiotetrazolylacetanilides with HIV-1 reverse

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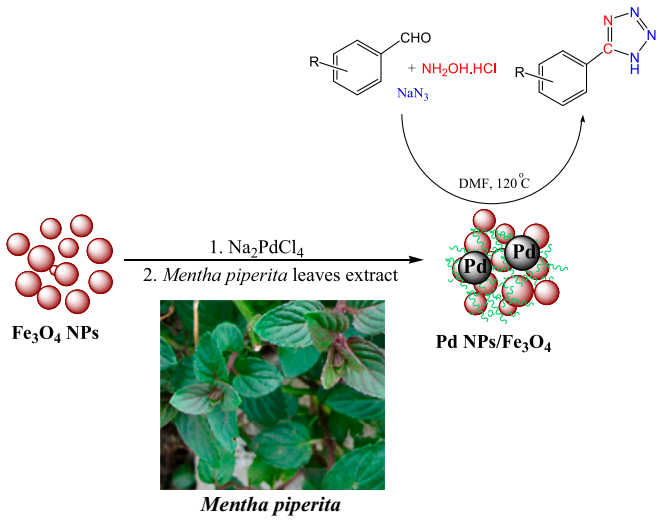


Scheme 1. Schematic bio-production of Pd NPs/Fe_3O_4 assisted by *Mentha piperita* leaves extract and its application for generation of 5-Phenyl-1*H*-tetrazoles.

transcriptase [7]. In the realm of medicinal chemistry, tetrazoles are viewed as reliable substitutes for carboxylic acids [8]. Additionally, these compounds are utilized in organo-metal science as efficient stabilizing agent for metal peptide frameworks, in peptide binding [9,10], and as ligands in coordination chemistry [11,12]. Moreover, tetrazoles are employed as plant growth enhancers, fungicides, and herbicides [13]. They also find applications in organocatalysis [14], specialty explosives [15], and photography [16].

Owing to their numerous benefits and diverse applicability, a variety of innovative synthrtic strategy for tetrazoles were actively pursued [17,18]. Among these, the [3 + 2] cycloaddition reaction between nitriles and NaN_3 is recognized as a standard method for producing 5-substituted 1*H*-tetrazoles. Typically, costly and hazardous phenylnitrile derivatives serve as the starting materials for tetrazole formation. Consequently, the intention to utilize more readily available substrates instead of nitriles and to employ inexpensive and accessible catalysts formed the basis for this research.

The *Mentha piperita* plant is important for many health reasons because it has special oils, the flavonoids and polyphenols which are present in its leaves extract [19]. Numerous studies have indicated its efficiency as antitumor, antiallergenic, antioxidant, and works well for fighting viruses, and bacteria [20]. Most research about *M. piperita* have looked at its essential oil, known as peppermint oil. The main ingredients in this oil are menthyl acetate, menthol, menthone, menthofuran, limonene, 1,8-cineole, bicyclogermacrene, pulegone, and sabinene [20]; as well as polyphenols, flavonoid glycoside and luteolin-diglucoronide are found in *Mentha piperita* [21].

Nowadays, catalysts have an important application in different fields [22–26]. Especially they are used for synthesis of different materials [27–30]. On the other hand, nanotechnology has brought about a major revolution in industry and daily life [31–33]. Today, nanomaterials have wide applications in various fields, including medicine, industry, the environment, etc. [34–37]. Structural manipulation or addition of different molecules, metals or ions to the surface of nanomaterials tailor their properties [38–43]. In the ongoing exploration of our studies on the synthesis of novel nanocatalyst for organic synthesis, we demonstrate here a novel approach for deposition of Pd nanoparticles over Fe_3O_4 nanoparticles mediated by *Mentha piperita* leaves extract (Pd NPs/Fe_3O_4), as depicted in Scheme 1. Following the characterization of prepared nanocomposite, its catalytic activity was investigated for preparing tetrazole derivatives. This method involves a one-pot [3 + 2] cycloaddition condensation between sodium azide, hydroxylamine hydrochloride and diverse aldehydes, with Pd NPs/Fe_3O_4 acting as an efficient catalyst (Scheme 1). The important point of the described strategy for synthesizing tetrazoles is employing aldehydes instead of nitriles in a tandem reaction.

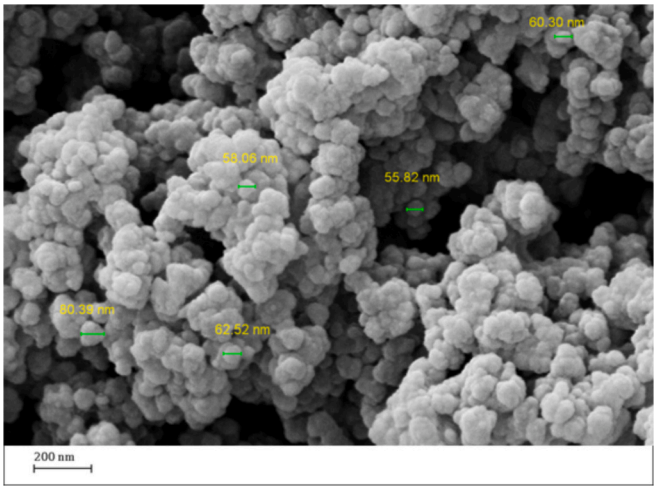


Fig. 1. FE-SEM image of Pd NPs/Fe_3O_4.

2. Experimental

2.1. Taking of Mentha piperita leaves extract

Fresh *Mentha piperita* leaves were provided from a garden in Nasr city, Egypt, and well cleaned with pure water and then vacuum dried at 50 °C. The dried leaf of *Mentha piperita* (1 g) were added to 20 mL of distilled H_2O and stand on a hot-plate at 80 °C for 30 min, and then permitted to cool and filtered for the subsequent step.

2.2. Fabrication of the Pd NPs/Fe_3O_4 catalyst

Recently, different NPs have been applied as nanocatalyst in synthesis of different compounds [44–47]. The Fe_3O_4 NPs were generated using a well-known procedure reported in previous research [48]. Initially, 200 mg of the produced Fe_3O_4 NPs was added in 50 mL of water and subjected to sonication for half an hour time. Then, 30 mg Na_2PdCl_4 which dissolved in 10 mL H_2O, was included drop-by-drop to the magnetic suspension, and stirring for 20 min. Then, 10 mL of *Mentha piperita* leaf extract was added, and the mixture was refluxed for 90 min. Following this period, the obtained Pd NPs/Fe_3O_4 were collected by a magnetic field, washed, and vacuum-dried at 50 °C. The concentration of Pd in the desired material was determined to be 0.073 mmol/g via ICP-OES data.

2.3. Pd NPs/Fe_3O_4 catalyzed generation of 5-phenyl-1H-tetrazoles

A combination of NaN_3 (1.0 mmol), $NH_2OH.HCl$ (1.0 mmol), aldehyde (1.0 mmol), and a catalytic amount of Pd NPs/Fe_3O_4 (0.3 mol%) in DMF (3 ml) were combined and stirred at 120 °C for an required duration (Table 2). Upon finishing of the process, as confirmed by TLC, the related catalyst was magnetically separated and the complete reaction mixture was filtered. Subsequently, 15 ml distilled water was added, and the desire tetrazole were extracted with EtOAc (3 × 15 ml). Then, 5 g dry Na_2SO_4 was added collected to the organic phase for drying it. After evaporation of the solvent, the crude solid was recrystallized in n-hexane/ethyl acetate (1:1) to yield high-purity products.

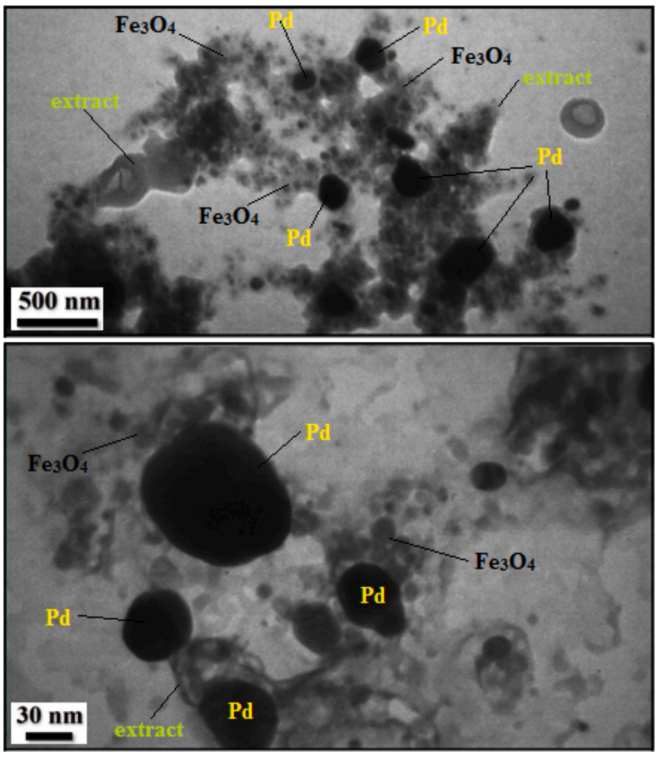


Fig. 2. TEM images of Pd NPs/Fe_3O_4.

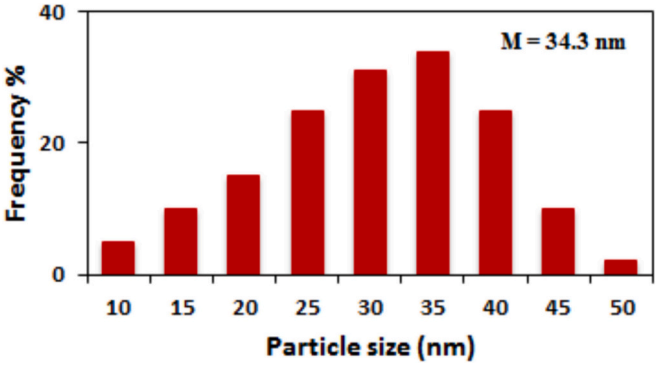


Fig. 3. Particle size histogram of Pd NPs.

3. Results and discussion

3.1. Characterization of Pd NPs/Fe_3O_4

Following the fabrication of the Pd NPs/Fe_3O_4 nanomaterial (Scheme 1), its structural characteristics were analyzed using a range of advanced techniques, including TEM, FE-SEM, elemental mapping, XRD, EDX, VSM, and ICP-OES.

The shape and dimensions of the produced Pd NPs/Fe_3O_4 nanoparticles were observed by TEM and FE-SEM analysis (Fig. 1 and Fig. 2). FE-SEM result indicates that the configuration of Pd NPs/Fe_3O_4

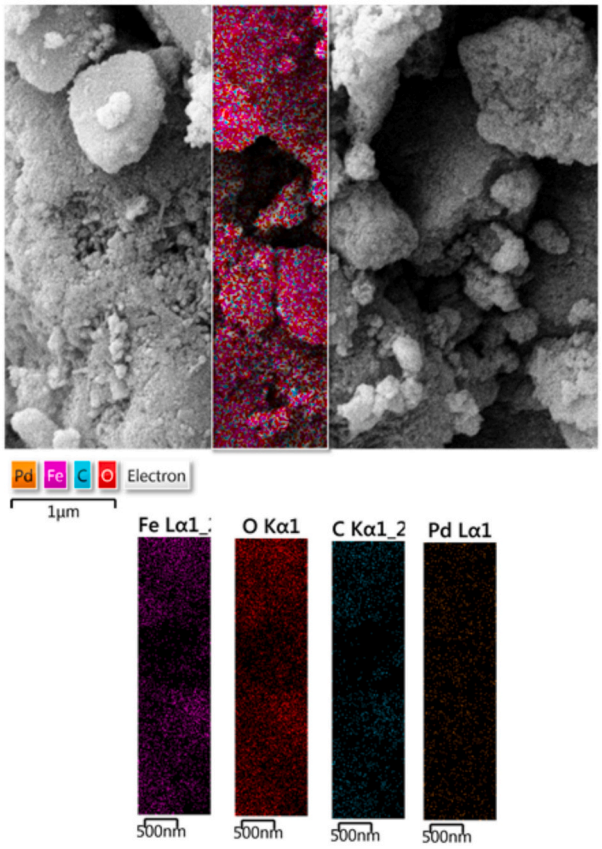


Fig. 5. EDX-elemental mapping of Pd NPs/Fe_3O_4.

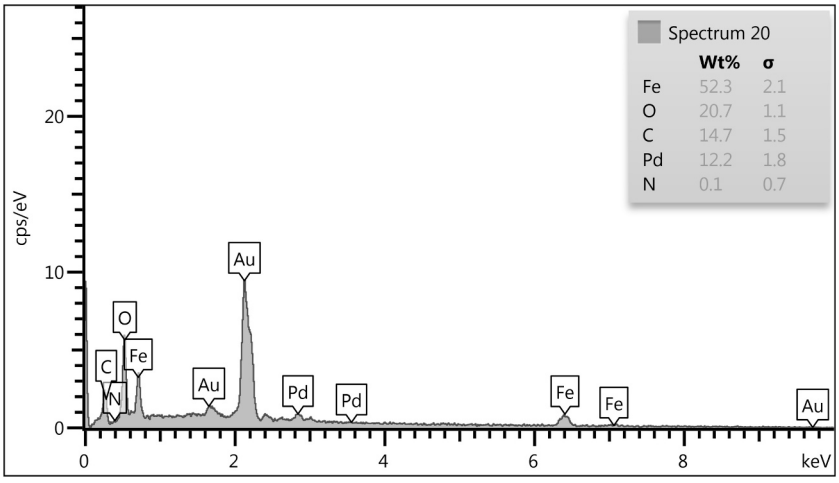


Fig. 4. EDX spectrum of Pd NPs/Fe_3O_4.

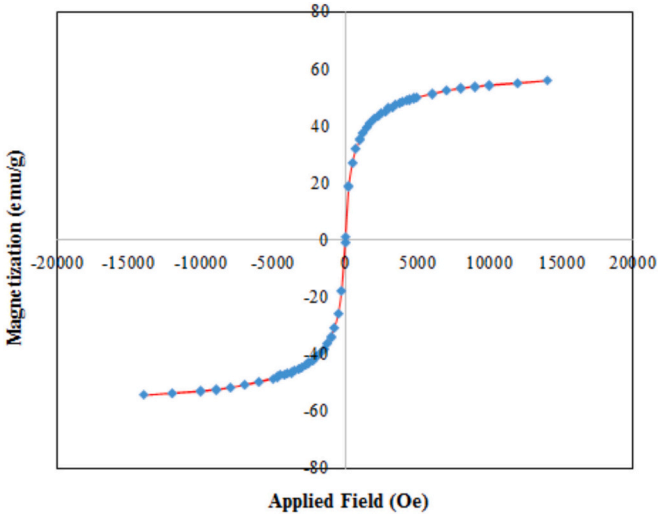
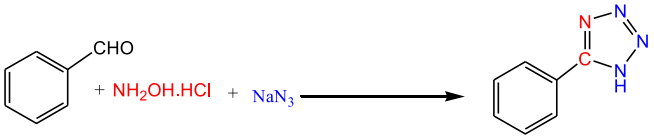


Fig. 6. VSM curve of Pd NPs/Fe_3O_4.

Table 1
Optimized experiments for the generation of tetrazole from benzaldehyde.[a]



Entry	Cat. (mol%)	Solvent	T (°C)	Time (h)	Yield (%)[b]
1	Pd NPs/Fe_3O_4 (0.3)	H_2O	reflux	12	0
2	Pd NPs/Fe_3O_4 (0.3)	EtOH	reflux	12	0
3	Pd NPs/Fe_3O_4 (0.3)	CH_3CN	reflux	12	0
4	Pd NPs/Fe_3O_4 (0.3)	CH_3Cl	reflux	12	0
5	Pd NPs/Fe_3O_4 (0.3)	DMSO	120	12	50
6	Pd NPs/Fe_3O_4 (0.3)	DMF	120	10	90
7	Pd NPs/Fe_3O_4 (0.3)	DMF	130	10	90
8	Pd NPs/Fe_3O_4 (0.3)	DMF	110	10	80
9	Pd NPs/Fe_3O_4 (0.3)	DMF	25	12	0
10	Pd NPs/Fe_3O_4 (0.2)	DMF	120	12	70
11	Pd NPs/Fe_3O_4 (0.4)	DMF	120	10	90
12	–	DMF	120	12	0
13	Fe_3O_4 (10 mg)	DMF	120	12	0
14	Fe_3O_4/Extract (10 mg)	DMF	120	12	0
15	Extract (10 mg)	DMF	120	12	0

[a] Reaction conditions: Benzaldehyde (1 mmol), $NH_2OH.HCl$ (1 mmol), NaN_3 (1 mmol) and Pd NPs/Fe_3O_4 (0.3 mol%) in solvent (3.0 mL).
[b] Isolated yield.

nanocomposite is roughly spherical shape with uniform dimension (Fig. 1). Fig. 2 presents the TEM image of the Pd NPs/Fe_3O_4, revealing that the nanoparticles are quasi spherical with a range dimension between 20 and 30 nm. Fig. 3 presented a particle size histogram, which illustrated that the average size of the Pd NPs was 34.3 nm.

EDX analysis was employed to examine the chemical makeup of the F Pd NPs/Fe_3O_4 nanocomposite. The existence of Pd in the matrix was also confirmed by the EDX technique connected to FE-SEM, alongside the elements C, N, O, and Fe (Fig. 4).

Furthermore, a significant study was conducted using elemental mapping alongside the FE-SEM image, offering valuable information regarding the elements' composition and their distributions in the matrix as colored dots (Fig. 5). The pattern of these dots signifies the dispersal of elemental species such as Fe, Pd, C, and O, which are distributed quite evenly.

The VSM assessment of the Pd NPs/Fe_3O_4 composite indicated a saturation magnetization (Ms) of 54.6 emu/g (Fig. 6), implying that the nanocomposite retains superparamagnetic characteristics even post-modification.

3.2. Catalytic performance of Pd NPs/Fe_3O_4 for generation of 5-phenyl-1H-tetrazoles

The capability of Pd NPs/Fe_3O_4 to act as a catalyst in the generation of tetrazole through a three-component and one-pot [3 + 2] cycloaddition process, involving sodium azide, hydroxylamine hydrochloride, and diverse aldehyde, was examined. To determine the proper reaction parameters, a model reaction was studied, employing hydroxylamine hydrochloride, benzaldehyde, and NaN_3 with Pd NPs/Fe_3O_4 as the catalyst. Different parameters including type of solvent, catalyst quantity and reaction temperature were analyzed, and the outcomes is documented as Table 1.

First, different solvents were assessed for the synthesis of 5-Phenyl-1*H*-tetrazole. There was no any product generation when the process was conducted in EtOH, H_2O, CH_3Cl, or CH_3CN (Table 1, entries 1–4). However, performing the model reaction in DMSO at 120 °C, a moderate product yield was obtained, as shown in Table 1, entry 5. The choice of solvent significantly influenced this process, with dimethylformamide (DMF) proving as the optimal solvent for achieving the best yields of the targeted product (Table 1, entry 6). Following this, we examined the effect of temperature in the process (Table 1, entries 6–9). Increasing of the temperature from 120 to 130 °C, did not substantially affect the yield or reaction duration (Table 1, entry 7). Conversely, reducing the temperature to 110 °C resulted in a decreased product yield (80%) (Table 1, entry 8). Additionally, no reaction happened at 25 °C, even with the present of catalyst (Table 1, entry 9).

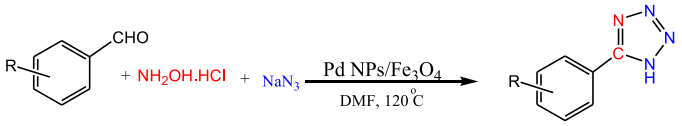
Subsequently, various quantities of catalyst were examined to determine the optimal rraction conditions. The yield decreased to 70% when the catalyst amount was droped to 0.2 mol% from 0.3 mol% (Table 1, entry 10). Additionally, increasing in the catalyst dosage did not lead to a significant enhance in product yield (Table 1, entry 11). Furthermore, without the Pd NPs/Fe_3O_4, no product was achieved (Table 1, entry 12). It is noteworthy to find the presence of Pd was vital for substrate conversion, as the Magnetic Fe_3O_4 NPs did not catalyze the model reaction.

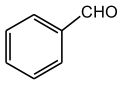
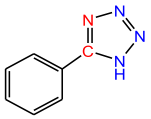
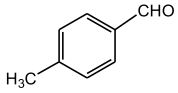
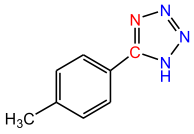
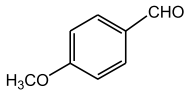
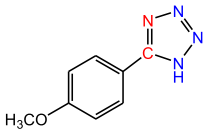
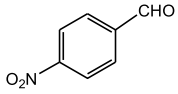
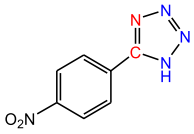
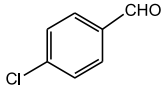
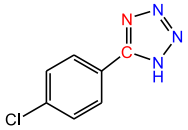
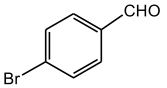
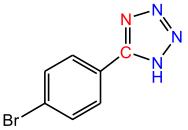
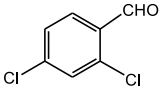
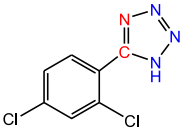
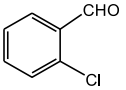
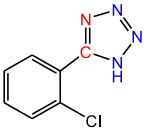
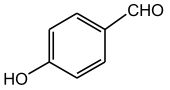
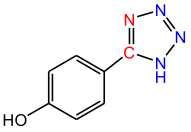
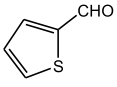
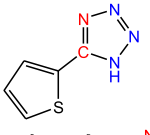
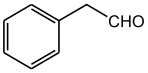
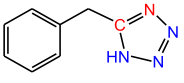
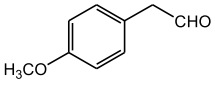
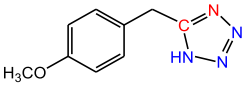
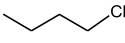
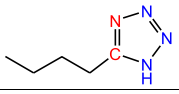
To assess the broad applicability of this approach, the reaction involving various aldehydes with numerous substituents on the aromatic ring were examined using the Pd NPs/Fe_3O_4 catalyst under refined conditions. As shown in Table 2, the results indicated that the reaction exhibits considerable generality and accommodating a wide range of substituted benzaldehydes. All reactions were completed in under 12 h, with tetrazole derivatives obtained in good yields (80–92%). Overall, both electronic and steric modifications have not notable influence on the products yield. The refined reaction conditions were also examined for different aliphatic aldehydes to obtain the moderate yields of the related tetrazoles (Table 2, entries 11–13).

To assess the catalyst's reusability, the condensation of benzaldehyde, $NH_2OH.HCl$ and NaN_3 was examined under the optimal system. Once the reaction was finished, the related catalyst was isolated from the medium using a magnet, rinsed with ethyl acetate and drying it for next cycle. As illustrated in Fig. 7, six cycles of catalyst recoveries occurred with minimal loss in catalytic performance. Furthermore, the catalyst's true heterogeneity was studied through a hot filtration experiment. At the midpoint (4 h, 56%), the catalyst was magnetically removed from the medium and allowed to continue reacting for an additional 4 h under those conditions. No notable rise in the product yield was detected, which affirmed the heterogeneous characteristics of the produced promoter. Additionally, the ICP study has showed the recyclability of the Pd NPs/Fe_3O_4 catalyst with negligible release of palladium species from the catalyst. Based on the ICP data, the amount of leached palladium from Pd NPs/Fe_3O_4 is 0.0019% after 6 consecutive times which is more negligible.

The proposed pathway [49–53] for creating 5-Phenyl-1*H*-tetrazoles from aryl aldehydes in the presence of Pd NPs/ Fe_3O_4 as the catalyst is

Table 2
Pd NPs/Fe_3O_4 catalyzed generation of 5-Phenyl-1*H*-tetrazoles.[a]



Entry	Aryl aldehyde	Product	Time (h)	Yield (%)[b]
1			8	92
2			9	90
3			9	92
4			10	90
5			10	88
6			12	85
7			12	85
8			12	80
9			10	85
10			12	80
11			14	60
12			15	65
13			24	50

[a] Reaction conditions: Pd NPs/Fe_3O_4 (0.3 mol%), $NH_2OH.HCl$ (1 mmol), (1 mmol), aldehyde (1 mmol) in DMF (3.0 mL) at 120 °C; [b]Isolated yield.

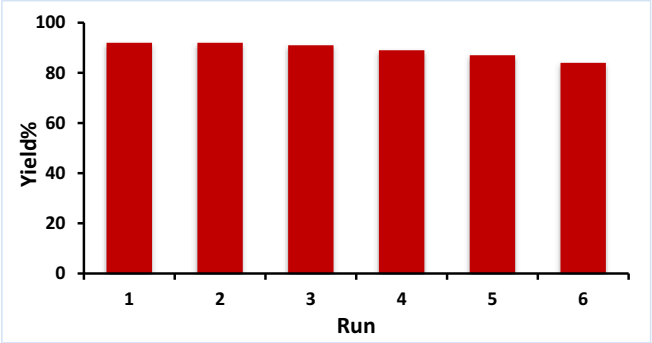
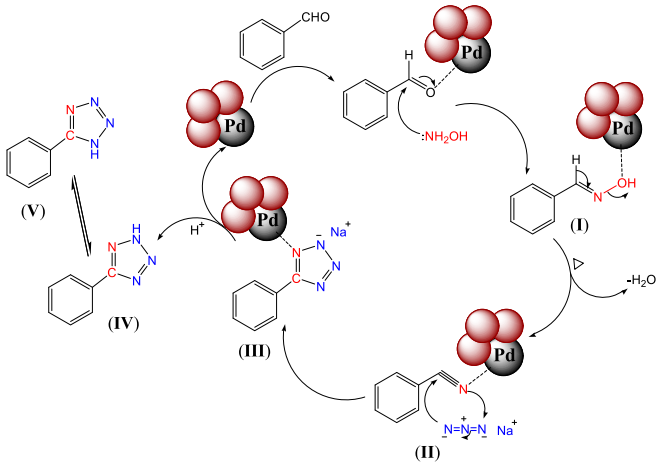


Fig. 7. The recycling of the Pd NPs/Fe_3O_4 catalyst for the model reaction.



Scheme 2. Proposed pathway for the generation of 5-Phenyl-1*H*-tetrazoles from aryl aldehydes in the presence of Pd NPs/Fe_3O_4.

illustrated in Scheme 2. Initially, an oxime is created from the nucleophilic attack of hydroxylamine to carbonyl group of aldehyde, which activated by Pd catalyst. Following this, water is eliminated to produce the nitrile product. Subsequently, the nitrile group is activated by Pd NPs, which enhances its cycloaddition with azide ion, leading to the formation of the intermediate **III**. After isolation of the nanocatalyst via a magnet and acidic treatment, both **IV** and **V** tautomers are generated. The more stable tautomer **V** (5-Phenyl-1*H*-tetrazole) is regarded as the desired compound.

4. Conclusions

In summary, this investigation demonstrates the green fabrication of palladium NPs affixed to the surface of Fe_3O_4 nanoparticles, utilizing *Mentha piperita* leaf extract in an aqueous solution. After the well production, various analytical methods were utilized to assess the properties of the created Pd NPs/Fe_3O_4 catalyst. In terms of catalysis, the obtained Pd NPs/Fe_3O_4 was evaluated for its function in the one-pot synthesis of tetrazoles from condensation of aromatic aldehydes, hydroxylamine hydrochloride, with sodium azide. The significant benefit of this approach is the substitution of harmful nitrile sustrates with available aldehydes. The Pd NPs/Fe_3O_4 catalyst was magnetically recovered and demonstrated the ability to be reused over 6 cycles without any notable decrease in its performance.

CRediT authorship contribution statement

Narinderjit Singh Sawaran Singh: Investigation, Funding acquisition, Formal analysis. **Ibrahim Saeed Gataa:** Funding acquisition, Formal analysis. **Maher Ali Rusho:** Methodology, Investigation, Funding acquisition, Formal analysis. **Pradeep Kumar Singh:** Methodology, Funding acquisition. **A.M.A. Mohamed:** Investigation, Funding acquisition, Formal analysis. **Akmal Abilkasimov:** Investigation, Funding acquisition. **Mutabar Latipova:** Funding acquisition, Formal analysis. **M.A. Diab:** Writing – review & editing, Writing – original draft, Supervision, Project administration. **Heba A. El-Sabban:** Writing – original draft, Supervision, Methodology. **Amir Ibrahim Ali Arabi:** Resources, Methodology, Investigation. **Saiful Islam:** Funding acquisition, Formal analysis.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Authorship statement

All persons who meet authorship criteria are listed as authors, and all authors certify that they have participated sufficiently in the work to take public responsibility for the content, including participation in the concept, design, analysis, writing, or revision of the manuscript. Furthermore, each author certifies that this material or similar material has not been and will not be submitted to or published in any other publication before its appearance in the *Inorg Chem Commun.*

Data availability

Data will be made available on request.

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