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Full Paper

Simple and Practical Synthesis of Various New Nickel Boride-Based Nanocomposites and their Applications for the Green and Expeditious Reduction of Nitroarenes to Arylamines under Wet-Solvent-Free Mechanochemical Grinding

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In this paper, we report a simple synthesis of four new nickel boride-based nanocomposites, namely $Ni_2B@ZrCl_4$, $Ni_2B@Cu_2O$, $Ni_2B@CuCl_2$ and $Ni_2B@FeCl_3$, from commercially available and cheap starting materials. All of the new Ni_2B-based nanocomposites were well characterized by Fourier-transform infrared spectroscopy, X-ray diffraction, scanning electron microscopy, and energy-dispersive X-ray spectroscopy. Further, the catalytic applications of these new nanocomposites were successfully evaluated in the wet-solvent-free reduction of aromatic nitro compounds to arylamines with sodium borohydride ($NaBH_4$) at room temperature by a mechanochemical grinding technique. All the introduced catalytic systems provide excellent yields of arylamines in very short reaction times for a wide range of substrates. Also, recoverability and reusability of the new nanocomposites were investigated.

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Introduction

At the beginning of the new century, green chemistry was recognized as a valuable culture and protocol for achieving sustainable development.[1] One of the important and effective aspects of green chemistry is solvent choice.[2] Solvents are often the largest sources of waste in chemical synthesis. Eliminating the use of solvents can significantly reduce the amount of waste and volatile organic compounds that are produced in a reaction process. Therefore, it can be said that the best solvent is no solvent.[3] In recent years, the technique of mechanochemical solid-state grinding has been recognized as an important and exceptional green solvent-free technique in organic synthesis not only for its solvent-free conditions but also for its time efficiency, cleanliness, and safer reaction profile, easy handling, high selectivity, and simple workup procedures.[4] Notably, it is carried out easily in the absence of or with minimal use of solvents in a mortar and pestle. Recently, various organic transformations have been accomplished using the solid-state grinding technique including aldol and cross-aldol condensation,[5] Biginelli reaction,[6] cyclopropanation,[7] Dieckmann condensation,[8] Grignard reaction,[9] Hantzsch dihydropyridine (DHP) synthesis,[10] synthesis of various heterocyclic compounds,[11] spiro compounds,[12] benzene rings,[13] and β-aminobutyric acids,[14] Knoevenagel condensation,[15] Reformatsky reaction,[16] Michael reaction,[17] Passerini reaction,[18] regioselective conversion of epoxides to vicinal hydroxythiocyanates,[19] synthesis of aryl toluenesulfonhydrazides and aryl toluenesulfonates,[20] synthesis of 1,3-dithiane and 1,3-dithiolane derivatives,[21] preparation of 5-aryl-2-furoyl-substituted thioureas and thiosemicarbazides,[22] synthesis of 2,6-dicyanoanilines,[23] one-pot synthesis of 3-amino-2,4-dicarbonitrile-5-methylbiphenyl derivatives,[24] preparation of 2-arylidene indan-1,3-diones,[25] preparation of 5-arylidene barbituric acid derivatives,[26] synthesis of water-soluble [60]fullerenol,[27] selective protection of primary alcohols and phenols,[28] synthesis of hexaalkoxytriphenylenes via oxidative coupling trimerization of 1,2-dialkoxybenzenes,[29] synthesis of 1-aryloxyacetyl-4-(2-benzofuroyl)semicarbazides,[30] facile synthesis of dialkyl disulfides,[31] synthesis of trisubstituted methanes,[32] preparation of azo dyes,[33] oxidation of oximes,[34] and reduction.[35] The catalyst is another important aspect of green chemistry.[36] Nowadays, the design and development of new nano-based catalysts attract a great deal of attention in synthetic organic chemistry because most of them have fewer drawbacks than classical catalysts, such as difficulties in purification of the final products, and deactivation of the catalyst.[37] Metal nanoparticles are undoubtedly among the most widely studied systems in modern nanoscience. Notably, this is because metals often have totally different properties when dispersed at nanometre dimensions. As an example, gold nanoparticles are active catalysts for oxidation reactions whereas the bulk metal is inactive. It should also be mentioned

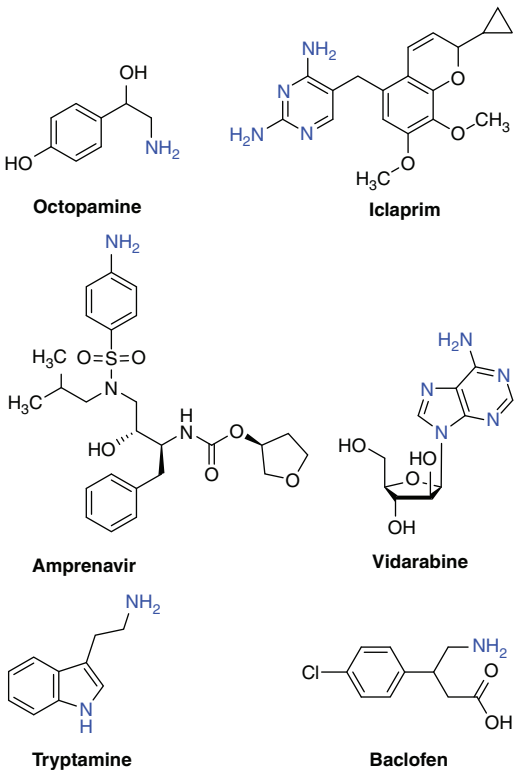


Fig. 1. Examples of amine-containing drugs on the market.

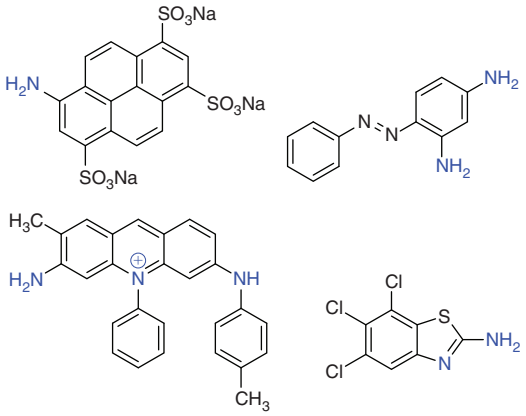
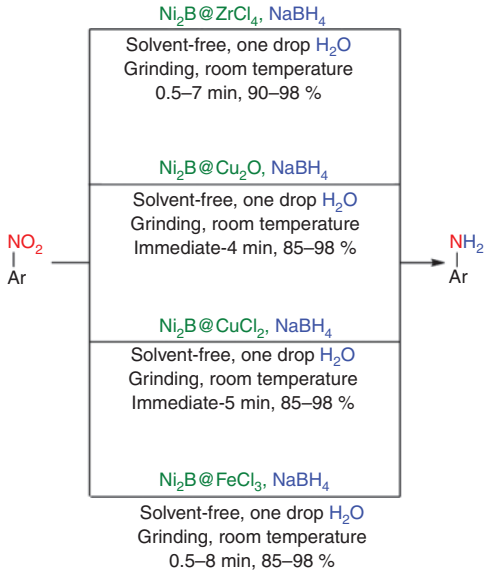


Fig. 2. Chemical structure of some organic dyes bearing amine functional groups.

that earth-abundant transition-metal-based nanoparticles are used as efficient and practical catalysts in various organic reactions because transition metal nanoparticle species not only mimic metal surface activation and catalysis but are also utilized as nano-sized catalyst supports.[38]

Amines are an extremely important family of compounds in chemical and pharmaceutical sciences. They are increasingly present in the chemical structure of a very large number of drugs (Fig. 1), dyes (Fig. 2), agrochemicals, polymers, and more.[39] Also, amines play a prominent role in synthetic chemistry as simple and efficient solvents[40] and catalysts.[41] Furthermore, 4-aminophenol oxalate salt is used as a corrosion inhibitor.[42] Amino groups can easily be replaced by other functional groups



Scheme 1. Reduction of nitroarenes to arylamines using wet-solvent-free mechanochemical grinding catalyzed by Ni_2B-based nanocomposites.

(such as H, F, Cl, Br, I, OH, CN, B, Sn, P, CF_3, SCF_3) through the corresponding diazonium salts.[43] One of the most important and straightforward methods for the preparation of aliphatic or aromatic amines is the reduction of nitro compounds. In recent years, numerous protocols for the reduction of nitro-containing compounds have been reported in the literature.[44] However, some of the reported methods have disadvantages including long reaction times, harsh reaction conditions, and use of toxic solvents and harmful catalysts. Owing to the high importance of amines and in response to the problems mentioned, designing new synthetic methods based on green chemistry protocols for the preparation of these valuable compounds is of great importance.

In continuation of our research program into the design and preparation of simple and cost-effective nanomaterials and using them as efficient catalysts in different types of organic reactions,[45] we report herein the convenient and affordable synthesis of the new Ni_2B-based nanocomposites $Ni_2B@ZrCl_4$, $Ni_2B@Cu_2O$, $Ni_2B@CuCl_2$, and $Ni_2B@FeCl_3$ and their impressive catalytic effects on the reduction of aromatic nitro compounds to arylamines with sodium borohydride ($NaBH_4$) at room temperature under wet-solvent-free mechanochemical grinding (Scheme 1).

Experimental

Reagents, Samples, and Apparatus

Chemicals were purchased from Merck, Fluka, and Sigma–Aldrich. Melting points were determined on an Electrothermal 9200 apparatus. Infrared spectra were recorded on a Nexus 670 Thermo Nicolet Fourier-transform infrared (FT-IR) spectrometer and measured as KBr discs. ^{1}H NMR spectra were recorded on a Bruker Avance spectrometer at 300 MHz in $CDCl_3$ with tetramethylsilane as internal standard. X-ray diffraction (XRD) measurements were done with a Philips PANalytical X'Pert Pro powder diffractometer. Particle morphology was examined using scanning electron microscopy (SEM) with a FESEM-Tescan MIRA3.

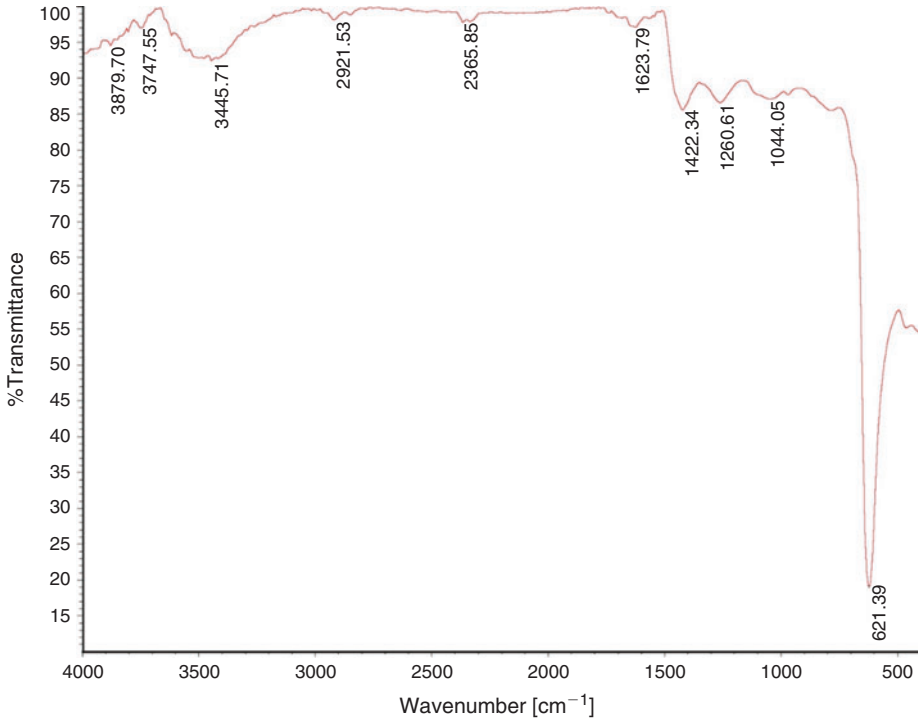


Fig. 3. FT-IR spectrum of $Ni_2B@Cu_2O$.

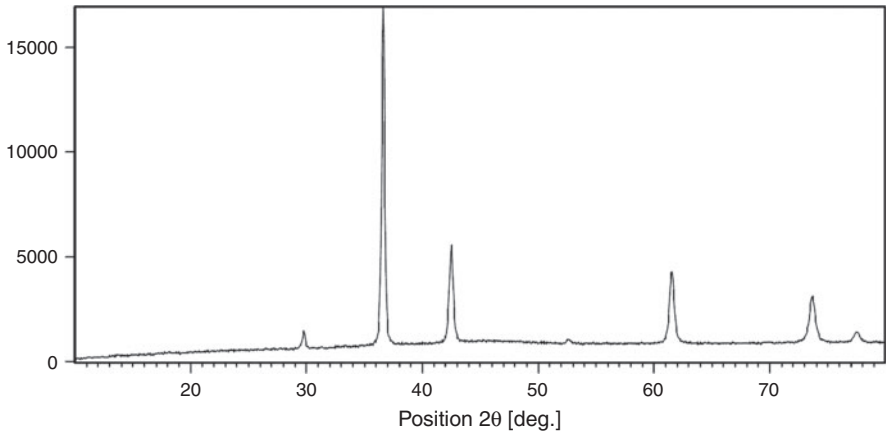


Fig. 4. XRD pattern of $Ni_2B@Cu_2O$.

Preparation of Nickel Boride (Ni_2B) Nanoparticles (NPs)

Nickel boride (Ni_2B) was prepared according to the literature.[46] In a dry two-necked round-bottomed flask (100 mL, equipped with a magnetic stirrer, $Ni(OAc)_2.4H_2O$ (1.244 g, 5 mmol) was dissolved in water (50 mL) under an atmosphere of nitrogen. Then, a solution of sodium borohydride (10 mL, 1 M) was gradually added within 30 s. The reaction is speedy and accompanied by the release of large amounts of hydrogen gas. On addition of the $NaBH_4$ solution, a black precipitate was produced. After complete removal of hydrogen gas from the reaction environment, a solution of sodium borohydride (5 mL, 1 M) was again gradually added to the reaction mixture under air. Finally, the black sediment was separated from the aqueous phase and washed with absolute ethanol (2 × 25 mL), and then was dried in air.

Typical Procedure for the Synthesis of Ni_2B-Based Nanocomposites

The preparation of $Ni_2B@ZrCl_4$ nanocomposite is presented here as a typical procedure. To prepare the $Ni_2B@ZrCl_4$ system, suspensions of $ZrCl_4$ (0.43 mmol, 100 mg) and Ni_2B (0.39 mmol, 50 mg) in dichloromethane (20 mL) were prepared separately. The $ZrCl_4$ suspension was then added slowly to the Ni_2B suspension and the mixture stirred vigorously for 30 min in a round-bottomed flask (100 mL) equipped with a magnetic stirrer. The dichloromethane was evaporated under reduced pressure and the residue was dried in air. $ZrCl_4$ was deposited on Ni_2B in a weight ratio of 100 : 50 respectively. The other three new nanocomposite systems viz, $Ni_2B@Cu_2O$, $Ni_2B@CuCl_2$, and $Ni_2B@FeCl_3$ were also synthesized according to this procedure.

General Procedure for the Reduction of Aromatic Nitro Compounds to Arylamines

As a representative example, a mixture of nitrobenzene (1 mmol), $Ni_2B@Cu_2O$ (54 mg), 1 drop of distilled water, and $NaBH_4$ (2.5 mmol) was ground using a simple porcelain mortar and pestle for 1 min at room temperature. After completion of the reaction (checked by TLC), distilled water (5 mL) was added to the reaction vessel, and subsequently the reaction mixture transferred to a round-bottom flask (25 mL) equipped with a magnetic stirrer. The mixture was stirred vigorously for 2 min. Next, the product was extracted with dichloromethane (5×3 mL). The extracts were dried with anhydrous sodium sulfate and then passed through a cotton filter. Evaporation of the solvent afforded pure aniline in 98 % yield.

Results and Discussion

Characterization of Nickel Boride (Ni_2B) NPs

We used FT-IR and XRD spectra and SEM images to characterize the Ni_2B nanoparticles (see Supplementary Material). Comparing these with spectra and images reported in the scientific literature confirmed the synthesis of Ni_2B nanoparticles.[47] The XRD spectrum of the prepared Ni_2B indicates an amorphous structure for the catalyst. The broad peak at 47.487° is associated with fine particles with nano dimensions. The amorphous morphology of the Ni_2B powder was also confirmed in SEM images. Stacked spherical particles were observed in the structures. Also, the SEM images indicated that the particles are in the nanometric range and the average particle size is 27 nm.

Characterization of New Nickel Boride-Based Nanocomposites

All of the isolated new nickel boride-based nanocomposites were characterized by FT-IR, XRD, SEM, and energy-dispersive X-ray (EDX) spectroscopy. (For spectra and images details, see Supplementary Material.) As an example, in this section, the structure of $Ni_2B@Cu_2O$ was investigated based on its spectra and related images. The FT-IR spectrum of $Ni_2B@Cu_2O$ (Fig. 3) shows an absorbance peak at 621 cm^{-1} due to Cu^I–O stretching, consistent with the literature.[48] The XRD spectrum (Fig. 4) contains seven peaks that are clearly distinguishable and broad. All of the diffraction peaks can be perfectly indexed to (110), (111), (200), (211), (220), (311), and (222) peaks of cubic Cu_2O (JCPDS nos 05–0667 and 78–2076).[49] The morphology and size of the prepared $Ni_2B@Cu_2O$ were characterized by SEM (Fig. 5). According to the SEM micrographs of the $Ni_2B@Cu_2O$ system, deformed cubic microcrystallites of Cu_2O have permeated into the aggregated structure of Ni_2B nanoparticles and made a nanocomposite in which Ni_2B NPs are scattered around the Cu_2O micro-crystallites. The high-magnification image (Fig. 5b) demonstrates that the prepared $Ni_2B@Cu_2O$ is composed of small nanoparticles. Additionally, EDX analysis from the $Ni_2B@Cu_2O$ nanocomposite system obtained (Fig. 6) showed the presence of the expected elements in the structure of the nanocomposite, namely boron, nickel, copper, and oxygen.

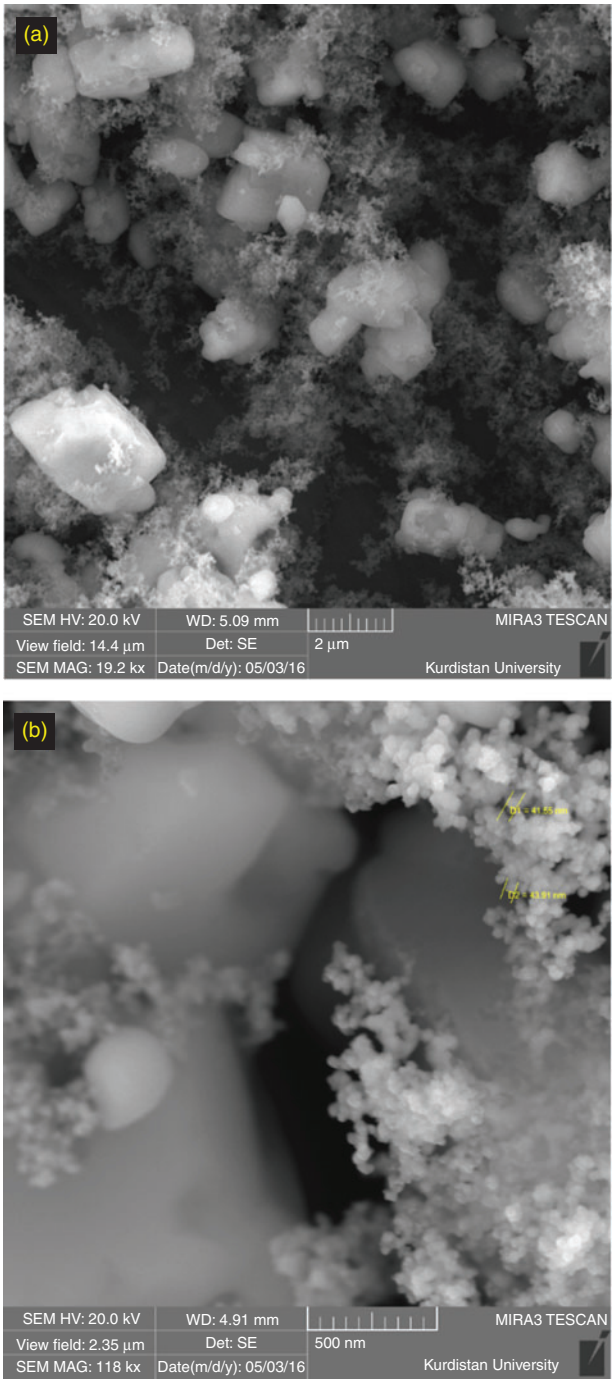


Fig. 5. SEM images of $Ni_2B@Cu_2O$.

Catalytic Activity

The catalytic activity of these new nanocomposites was tested in the reduction of aromatic nitro compounds to the corresponding arylamines. First, we chose the reaction of nitrobenzene with $NaBH_4$ in the presence of a catalytic amount of $Ni_2B@ZrCl_4$ as a model reaction. Then, we optimized the reaction conditions using various molar ratios of sodium borohydride as well as testing solvents such as methanol, ethanol, ethyl acetate, tetrahydrofuran (THF), acetonitrile, and water, and solvent-free conditions at room temperature and

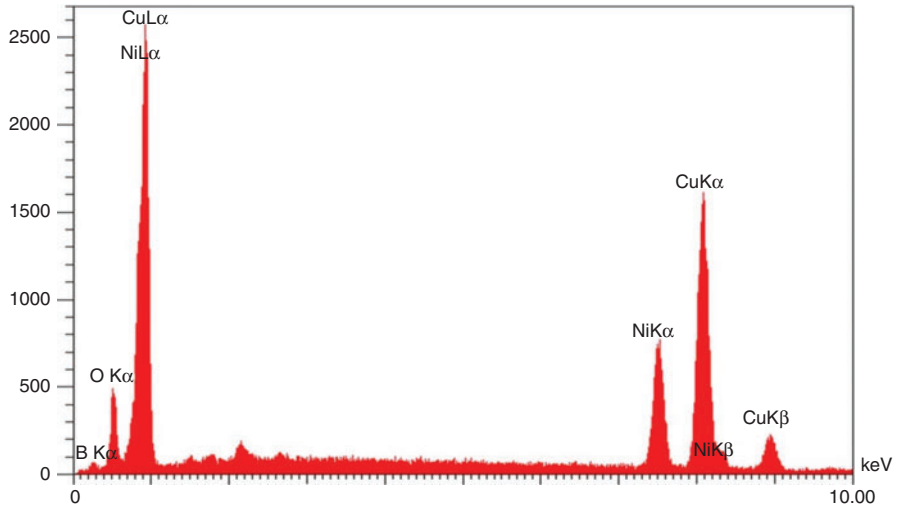
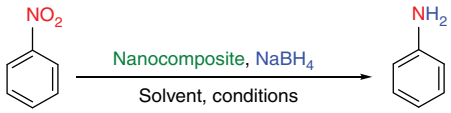


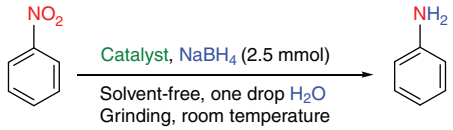
Fig. 6. EDX spectrum of $Ni_2B@Cu_2O$.

Table 1. Optimization of reaction conditions
rt, room temperature



Entry	$NaBH_4$ [mmol]	Nanocomposite [mg]	Solvent	Conditions	H_2O	Time [min]	Conversion [%]
1	3	$Ni_2B@ZrCl_4$ (72)	MeOH	Reflux	–	100	0
2	3	$Ni_2B@ZrCl_4$ (72)	EtOH	Reflux	–	100	0
3	3	$Ni_2B@ZrCl_4$ (72)	EtOAc	Reflux	–	100	0
4	3	$Ni_2B@ZrCl_4$ (72)	THF	Reflux	–	100	0
5	3	$Ni_2B@ZrCl_4$ (72)	CH_3CN	Reflux	–	100	0
6	3	$Ni_2B@ZrCl_4$ (72)	H_2O	Reflux	–	120	50
7	2	$Ni_2B@ZrCl_4$ (36)	–	Grinding, rt	1 drop	60	70
8	2.5	$Ni_2B@ZrCl_4$ (36)	–	Grinding, rt	1 drop	7	100
9	2.5	$Ni_2B@ZrCl_4$ (72)	–	Grinding, rt	1 drop	3	100
10	3	$Ni_2B@Cu_2O$ (54)	MeOH	Reflux	–	100	0
11	3	$Ni_2B@Cu_2O$ (54)	EtOH	Reflux	–	100	0
12	3	$Ni_2B@Cu_2O$ (54)	EtOAc	Reflux	–	100	0
13	3	$Ni_2B@Cu_2O$ (54)	THF	Reflux	–	100	0
14	3	$Ni_2B@Cu_2O$ (54)	CH_3CN	Reflux	–	100	0
15	3	$Ni_2B@Cu_2O$ (54)	H_2O	Reflux	–	120	60
16	2	$Ni_2B@Cu_2O$ (27)	–	Grinding, rt	1 drop	60	70
17	2.5	$Ni_2B@Cu_2O$ (27)	–	Grinding, rt	1 drop	5	100
18	2.5	$Ni_2B@Cu_2O$ (54)	–	Grinding, rt	1 drop	1	100
19	3	$Ni_2B@CuCl_2$ (52)	MeOH	Reflux	–	100	0
20	3	$Ni_2B@CuCl_2$ (52)	EtOH	Reflux	–	100	0
21	3	$Ni_2B@CuCl_2$ (52)	EtOAc	Reflux	–	100	0
22	3	$Ni_2B@CuCl_2$ (52)	THF	Reflux	–	100	0
23	3	$Ni_2B@CuCl_2$ (52)	CH_3CN	Reflux	–	100	0
24	3	$Ni_2B@CuCl_2$ (52)	H_2O	Reflux	–	120	60
25	2	$Ni_2B@CuCl_2$ (26)	–	Grinding, rt	1 drop	60	70
26	2.5	$Ni_2B@CuCl_2$ (26)	–	Grinding, rt	1 drop	6	100
27	2.5	$Ni_2B@CuCl_2$ (52)	–	Grinding, rt	1 drop	2	100
28	3	$Ni_2B@FeCl_3$ (58)	MeOH	Reflux	–	100	0
29	3	$Ni_2B@FeCl_3$ (58)	EtOH	Reflux	–	100	0
30	3	$Ni_2B@FeCl_3$ (58)	EtOAc	Reflux	–	100	0
31	3	$Ni_2B@FeCl_3$ (58)	THF	Reflux	–	100	0
32	3	$Ni_2B@FeCl_3$ (58)	CH_3CN	Reflux	–	100	0
33	3	$Ni_2B@FeCl_3$ (58)	H_2O	Reflux	–	120	60
34	2	$Ni_2B@FeCl_3$ (29)	–	Grinding, rt	1 drop	60	80
35	2.5	$Ni_2B@FeCl_3$ (29)	–	Grinding, rt	1 drop	10	100
36	2.5	$Ni_2B@FeCl_3$ (58)	–	Grinding, rt	1 drop	5	100

Table 2. Comparison of the catalytic activity of prepared nanocomposites with their component parts



Entry	Catalyst	Time [min]	Conversion [%]
1	Ni_2B (0.2 mmol)	60	50
2	$ZrCl_4$ (0.2 mmol)	60	5
3	Cu_2O (0.2 mmol)	60	5
4	$CuCl_2$ (0.2 mmol)	60	0
5	$FeCl_3$ (0.2 mmol)	60	5
6	Ni_2B (0.2 mmol) + $ZrCl_4$ (0.2 mmol)	60	55
7	Ni_2B (0.2 mmol) + Cu_2O (0.2 mmol)	60	65
8	Ni_2B (0.2 mmol) + $CuCl_2$ (0.2 mmol)	60	60
9	Ni_2B (0.2 mmol) + $FeCl_3$ (0.2 mmol)	60	60
10	Ni_2B@$ZrCl_4$ (72 mg)	3	100
11	Ni_2B@Cu_2O (54 mg)	1	100
12	Ni_2B@$CuCl_2$ (52 mg)	2	100
13	Ni_2B@$FeCl_3$ (58 mg)	5	100

reflux. When the reaction was carried out with 2.5 mmol of $NaBH_4$ and one drop of water in the presence of Ni_2B@$ZrCl_4$ (72 mg) under solvent-free reaction conditions using a mechanochemical grinding technique (Table 1, entry 9), the best result was achieved in a short time of 3 min. We repeated all the optimization steps (mentioned above) in the presence of the other new nanocomposites Ni_2B@Cu_2O, Ni_2B@$CuCl_2$, and Ni_2B@$FeCl_3$. Interestingly, we found that all the four new nanocomposites exhibited significant catalytic effects in the reduction of nitrobenzene to aniline under solvent-free reaction conditions at room temperature. It should also be mentioned that when the model reaction was attempted with Ni_2B, $ZrCl_4$, Cu_2O, $CuCl_2$ alone, and also $FeCl_3$ under the optimal reaction conditions, the nitrobenzene reduction process was very disappointing, in that after a fairly long time (60 min), the reaction was not complete (Table 2, entries 1–5). Also, we tested the model reaction using Ni_2B nanoparticles along with the aforementioned Lewis acids simultaneously as combined catalytic systems (Table 2, entries 6–9). Unfortunately, these two-component catalytic systems were also not suitable for the reduction of nitrobenzene. Therefore, as shown in Table 2, our prepared nanomaterials play a very important role in this reduction reaction. In the next step, with the optimal reaction conditions in hand, various types of aromatic nitro compounds including some with both electron-withdrawing (EWD) and electron-donating groups (EDG) were converted to the corresponding arylamines in excellent yields (Table 3). Notably, in some aromatic nitro compounds having acetyl or formyl substituents, reactions were typically complete, with reduction of both nitro and carbonyl functional groups. Amide (Table 3, entry 12) and dinitro- (Table 3, entry 19) groups and nitrophenyl hydrazine (Table 3, entry 20) or nitrophenyl carboxylic acid (Table 3, entry 21) did not undergo any change in the studied systems. It should be noted that no reduction reaction takes place without wet reaction conditions. Therefore, the presence of one drop of water inside the reaction medium is essential. It is very important to note that all of our efforts for the reduction of aliphatic nitro compounds using the current reduction systems failed (Table 3, entries 22 and 23). A plausible mechanism for the reduction of aromatic nitro compounds to arylamines with $NaBH_4$ in the presence of nickel boride-based nanocomposites is shown in Scheme 2.

Reusability of the Ni_2B-Based Nanocomposites

The recovery and reusability of a catalyst is a very important aspect in green catalytic processes. The recoverability and reusability of the prepared nanocomposites were investigated in this reduction reaction. After completion of the first reaction, we added water (5 mL) to the reaction medium. Next, the black solid was separated simply by filtration and then further washed with water and ethanol and dried at room temperature. After spectral analysis, it was found that the black powder obtained was Ni_2B. This means that the Lewis acid part of the nanocomposites was removed during the reaction process and/or during separation (after adding water). In the next step, we grafted fresh Lewis acids (namely $ZrCl_4$, Cu_2O, $CuCl_2$, and $FeCl_3$) on the recovered nickel boride. We observed no significant change in the activity of the rebuilt Ni_2B-based nanocomposites during the recycling experiment with fresh nitrobenzene under the optimized reaction conditions.

Comparative Study

To show the value, efficiency, and capability of the present green and expeditious protocols for the reduction of aromatic nitro compounds to the corresponding arylamines, they were compared with some previously reported methodologies. Results are summarized in Table 4. It is worth noting that the new protocols presented in the current paper are superior to many of the others in terms of catalyst loading, reaction time, cost-effectiveness, favourable yields of products, non-use of solvent, use of solvent-free mechanochemical grinding technique, and so on.

Conclusions

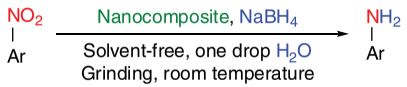
A series of new nickel boride-based nanocomposites, namely Ni_2B@$ZrCl_4$, Ni_2B@Cu_2O, Ni_2B@$CuCl_2$, and Ni_2B@$FeCl_3$, were prepared using a simple operation and characterized by FT-IR, XRD, SEM, and EDX. All the synthesized Ni_2B-based nanocomposites showed very satisfactory catalytic activity in the reduction of aromatic nitro compounds to the corresponding arylamines. The reaction occurs with $NaBH_4$ as a reducing agent under wet-solvent-free mechanochemical grinding. It is worthy of note that the reduction protocols described have significant advantages (especially from the standpoint of green chemistry and industrial chemistry), including very short reaction times, high yields of products, the use of nickel boride-based nanocomposites as very inexpensive new nanocatalysts, non-use of toxic solvents, and the use of a wet-solvent-free mechanochemical grinding technique.

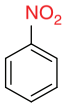
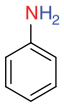
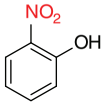
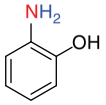
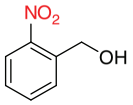
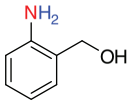
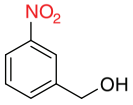
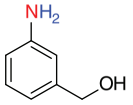
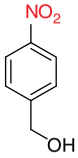
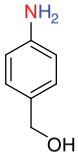
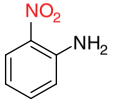
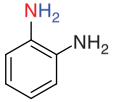
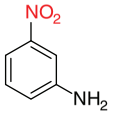
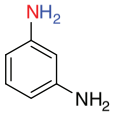
Supplementary Material

FT-IR, XRD, SEM, and EDX results of all Ni_2B-based nanocomposites along with FT-IR and SEM results of Ni_2B nanoparticles and also some selected FT-IR and H NMR spectra of prepared amines are available on the Journal's website.

Table 3. Green and expeditious reduction of nitroarenes to arylamines under wet-solvent-free mechanochemical grinding catalyzed by Ni_2B-based nanocomposites

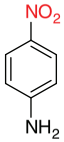
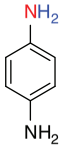
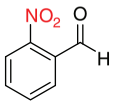
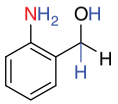
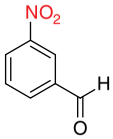
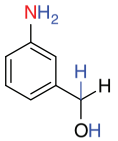
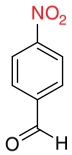
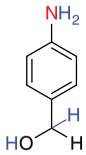
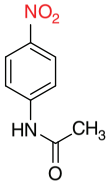
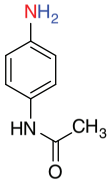
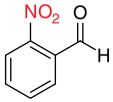
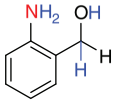
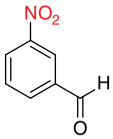
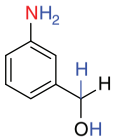
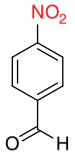
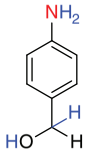
MR, molar ratio substrate/$NaBH_4$; Cat., catalyst; Imm, immediate; NR, no reaction

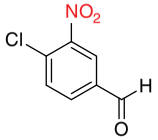
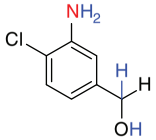
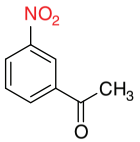
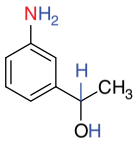
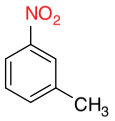
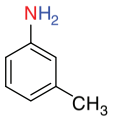
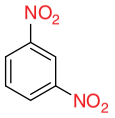
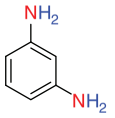
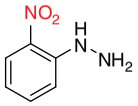
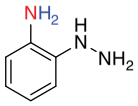
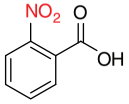
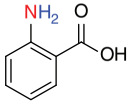
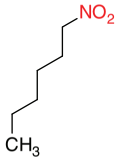
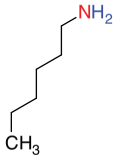
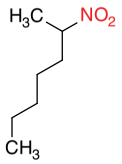
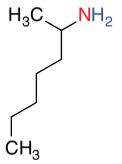


Entry	Substrate (nitroarene)	Product (arylamine)	Nanocomposite			
			$Ni_2B@ZrCl_4$	$Ni_2B@Cu_2O$	$Ni_2B@CuCl_2$	$Ni_2B@FeCl_3$
1			MR 1 : 2.5 Cat. 72 mg Time 3 min Yield[A] 98 %	MR 1 : 2.5 Cat. 54 mg Time 1 min Yield 98 %	MR 1 : 2.5 Cat. 52 mg Time 2 min Yield 98 %	MR 1 : 2.5 Cat. 58 mg Time 5 min Yield 98 %
2			MR 1 : 2.5 Cat. 72 mg Time 1 min Yield 97 %	MR 1 : 2.5 Cat. 54 mg Time Imm[D] Yield 97 %	MR 1 : 2.5 Cat. 52 mg Time 2 min Yield 97 %	MR 1 : 2.5 Cat. 58 mg Time 3 min Yield 97 %
3			MR 1 : 2.5 Cat. 72 mg Time 3 min Yield 94 %	MR 1 : 2.5 Cat. 54 mg Time 2 min Yield 94 %	MR 1 : 2.5 Cat. 52 mg Time 3 min Yield 94 %	MR 1 : 2.5 Cat. 58 mg Time 5 min Yield 94 %
4			MR 1 : 3 Cat. 72 mg Time 5 min Yield 96 %	MR 1 : 3 Cat. 54 mg Time 4 min Yield 96 %	MR 1 : 3 Cat. 52 mg Time 4 min Yield 96 %	MR 1 : 3 Cat. 58 mg Time 7 min Yield 96 %
5			MR 1 : 2.5 Cat. 72 mg Time 3 min Yield 94 %	MR 1 : 2.5 Cat. 54 mg Time 2 min Yield 94 %	MR 1 : 2.5 Cat. 52 mg Time 3 min Yield 94 %	MR 1 : 2.5 Cat. 58 mg Time 5 min Yield 94 %
6			MR 1 : 2.5 Cat. 72 mg Time 7 min Yield 90 %	MR 1 : 3 Cat. 54 mg Time 4 min Yield 87 %	MR 1 : 2.5 Cat. 52 mg Time 4 min Yield 87 %	MR 1 : 2.5 Cat. 58 mg Time 8 min Yield 87 %
7			MR 1 : 2.5 Cat. 72 mg Time 1 min Yield 94 %	MR 1 : 2.5 Cat. 54 mg Time Imm Yield 94 %	MR 1 : 2.5 Cat. 52 mg Time 1 min Yield 94 %	MR 1 : 2.5 Cat. 58 mg Time 3 min Yield 94 %

(*Continued*)

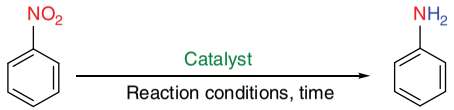
Table 3. (Continued)

Entry	Substrate (nitroarene)	Product (arylamine)	Nanocomposite			
			$Ni_2B@ZrCl_4$	$Ni_2B@Cu_2O$	$Ni_2B@CuCl_2$	$Ni_2B@FeCl_3$
8			MR 1 : 2.5 Cat. 72 mg Time 7 min Yield 92 %	MR 1 : 4 Cat. 54 mg Time 4 min Yield 85 %	MR 1 : 2.5 Cat. 52 mg Time 4 min Yield 85 %	MR 1 : 2.5 Cat. 58 mg Time 8 min Yield 85 %
9			MR 1 : 3 Cat. 72 mg Time 4 min Yield 94 %	MR 1 : 3 Cat. 54 mg Time 2 min Yield 92 %	MR 1 : 3 Cat. 52 mg Time 3 min Yield 92 %	MR 1 : 3 Cat. 58 mg Time 5 min Yield 92 %
10			MR 1 : 4 Cat. 72 mg Time 7 min Yield 97 %	MR 1 : 4 Cat. 54 mg Time 4 min Yield 97 %	MR 1 : 4 Cat. 52 mg Time 5 min Yield 97 %	MR 1 : 4 Cat. 58 mg Time 7 min Yield 97 %
11			MR 1 : 3 Cat. 72 mg Time 4 min Yield 92 %	MR 1 : 3 Cat. 54 mg Time 2 min Yield 92 %	MR 1 : 3 Cat. 52 mg Time 3 min Yield 92 %	MR 1 : 3 Cat. 58 mg Time 5 min Yield 92 %
12			MR 1 : 2.5 Cat. 72 mg Time 5 min Yield 95 %	MR 1 : 2.5 Cat. 54 mg Time 3 min Yield 95 %	MR 1 : 2.5 Cat. 52 mg Time 4 min Yield 95 %	MR 1 : 2.5 Cat. 58 mg Time 6 min Yield 95 %
13			MR 1 : 0.5 Cat. 18 mg Time 30 s Yield 98 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 96 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 96 %	MR 1 : 0.5 Cat. 14 mg Time 30 s Yield 96 %
14			MR 1 : 0.5 Cat. 18 mg Time 30 s Yield 94 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 92 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 92 %	MR 1 : 0.5 Cat. 14 mg Time 30 s Yield 92 %
15			MR 1 : 0.5 Cat. 18 mg Time 30 s Yield 96 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 94 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 94 %	MR 1 : 0.5 Cat 14 mg Time 30 s Yield 94 %

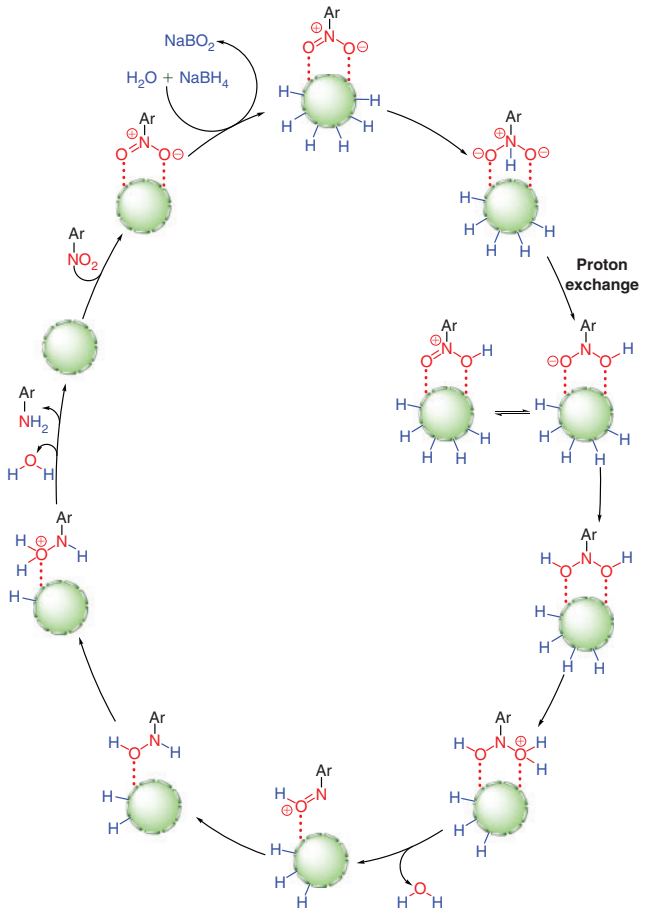
16			MR 1 : 0.5 Cat. 18 mg Time 30 s Yield 98 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 98 %	MR 1 : 0.5 Cat. 13 mg Time Imm Yield 98 %	MR 1 : 0.5 Cat. 14 mg Time 30 s Yield 98 %
17			MR 1 : 2 Cat. 36 mg Time 5 min Yield 92 %	MR 1 : 2 Cat. 27 mg Time 3 min Yield 90 %	MR 1 : 2 Cat. 26 mg Time 4 min Yield 90 %	MR 1 : 2 Cat. 29 mg Time 6 min Yield 90 %
18			MR 1 : 2.5 Cat. 72 mg Time 2 min Yield 97 %	MR 1 : 2.5 Cat. 54 mg Time 2 min Yield 97 %	MR 1 : 2.5 Cat. 52 mg Time 2 min Yield 97 %	MR 1 : 2.5 Cat. 58 mg Time 4 min Yield 97 %
19			MR 1 : 2.5 Cat. 72 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 54 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 52 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 58 mg Time 60 min Yield NR
20			MR 1 : 2.5 Cat. 72 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 54 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 52 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 58 mg Time 60 min Yield NR
21			MR 1 : 2.5 Cat. 72 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 54 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 52 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 58 mg Time 60 min Yield NR
22			MR 1 : 2.5 Cat. 72 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 54 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 52 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 58 mg Time 60 min Yield NR
23			MR 1 : 2.5 Cat. 72 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 54 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 52 mg Time 60 min Yield NR	MR 1 : 2.5 Cat. 58 mg Time 60 min Yield NR

[A]Yields refer to isolated pure products.

Table 4. Comparison of the reduction of nitrobenzene to aniline in the presence of the four new Ni_2B-based nanocomposites with other reported catalytic systems



Entry	catalyst	Reaction conditions	Time	Yield [%]	Reference
1	Cu-BTC@Fe_3O_4	EtOH/H_2O (3 : 1), 45°C, $NaBH_4$	3 h	99	[50]
2	NAP-Mg-Pd(0)	THF, rt, H_2 atmosphere	2 h	98	[51]
3	SiO_2-$Ag_{(Nano)}$	H_2O, reflux, $NaBH_4$	1 h	100	[52]
4	Ag NPs/$GR_{3.0}$-PAMAM	H_2O, rt, $NaBH_4$	20 min	100	[53]
5	Co-Mo_2C/AC	EtOH, 80°C, $NH_2NH_2.H_2O$	2 h	100	[54]
6	$CuBr_2$	EtOH, rt, $NaBH_4$	5 h	90	[55]
7	IO@Ni NPs	H_2O, rt, $NaBH_4$	25 min	95	[56]
8	Au/Al_2O_3	CH_3CN, LED, 80°C, HCOOH	2 h	100	[57]
9	GLRG 4	DI H_2O, 70°C, $NH_2NH_2.H_2O$	3 h	94	[58]
10	BNG-800	EtOH, 70°C, $NH_2NH_2.H_2O$	3 h	99	[59]
11	Ni-pol	EtOH/Et_2O (1 : 1), rt (under N_2), $NaBH_4$	2 h	90	[60]
12	IT-MHAp-Ag	H_2O, reflux, $NaBH_4$	25 min	98	[61]
13	Pd@CQD@Fe_3O_4	H_2O/EtOH (5 : 1), rt, $NaBH_4$	2 h	97	[62]
14	Cu-Ru/MWCNT	EtOH/H_2O (9 : 1), rt, $NaBH_4$	30 min	60	[63]
15	Pd-NPs@Oak Gum	EtOH/H_2O (1 : 2), 50°C, $NaBH_4$	1 h	96	[64]
16	Fe_3O_4@SiO_2/EP.EN.EG@Cu	H_2O, 50°C, $NaBH_4$	15 min	85	[65]
17	Pd/GYLPCO	H_2O, rt, $NaBH_4$	10 min	99	[66]
18	Ni/mZSM-5	H_2O, rt, $NaBH_4$	2 min	97	[67]
19	Go/Au	H_2O, rt, $NaBH_4$	35 h	92	[68]
20	Ag/MMT-K10	Isopropanol, KOH, rt	2.5 h	98	[69]
21	Mag-IL-Pd	H_2O, 90°C, HCO_2NH_4	15 h	99	[70]
22	GA-Pd/ZnO	MeOH, rt, H_2 atmosphere	2 h	98	[71]
23	GA-Pd/TiO_2	MeOH, rt, H_2 atmosphere	2 h	99	[71]
24	Fe_3O_4/SiO_2/CdS	Isopropanol, blue LED (3 W)	20 h	43	[72]
25	Fe_3O_4@BRAC	Isopropanol, KOH, MW (300 W), 85°C	15 min	99	[73]
26	Pd@SBA-15/TET	H_2O, rt (under N_2), $NaBH_4$	40 min	98	[74]
27	Cu_2O	EtOH, 30°C, NH_3BH_3	25 min	92	[75]
28	Fe-Cu@MCC	H_2O, 70°C, $NaBH_4$	8 min	93	[45b]
29	$NiFe_2O_4$@Cu NPs	H_2O/EtOH (1.5 : 0.5), reflux, $NaBH_4$	1 min	95	[45c]
30	Fe_3O_4@APTMS@$ZrCp_2$	H_2O, rt, glycerol	40 min	96	[45e]
31	Fe_3O_4@$Cu(OH)_x$ NPs	H_2O, 55–60°C, $NaBH_4$	3 min	95	[45g]
32	Cu NPs	H_2O, 80°C, $NaBH_4$	4 min	91	[45h]
33	Ni_2B	H_2O, rt, $NaBH_4$	3 min	95	[45i]
34	Ni_2B@$ZrCl_4$	Solvent-free, grinding, one drop H_2O, rt, $NaBH_4$	3 min	98	Present work
35	Ni_2B@Cu_2O	Solvent-free, grinding, one drop H_2O, rt, $NaBH_4$	1 min	98	Present work
36	Ni_2B@$CuCl_2$	Solvent-free, grinding, one drop H_2O, rt, $NaBH_4$	2 min	98	Present work
37	Ni_2B@$FeCl_3$	Solvent-free, grinding, one drop H_2O, rt, $NaBH_4$	5 min	98	Present work



Scheme 2. Plausible mechanism for the reduction of aromatic nitro compounds to arylamines with $NaBH_4$ in the presence of nickel boride-based nanocomposites.

Conflict of interest

The authors declare no conflicts of interest.

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