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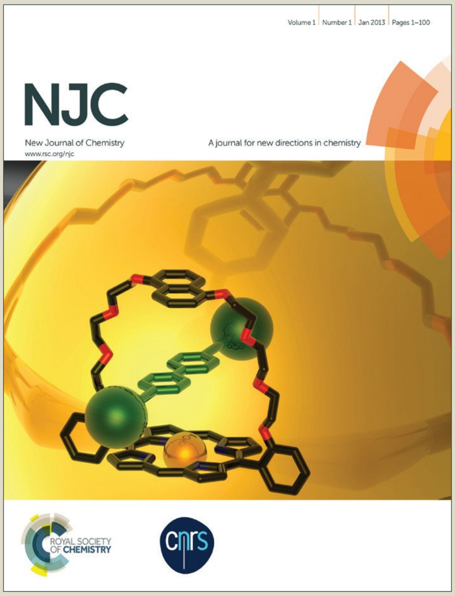

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PAPER

Green synthesis and characterization of silver nanoparticles using *Ferula Latisecta* leaf extract and its application as catalyst for the safe and simple one-pot preparation of spirooxindoles in water

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The present study reports an environmentally friendly and green synthetic method for the preparation of silver nanoparticles (Ag NPs) using *Ferula Latisecta* leaf extract which acts as a reducing and capping agent that can reduce silver ions into AgNPs without using any harsh conditions. In addition, these nanoparticles were found to be excellent catalytic activity in simple and safe one-pot preparation of spirooxindoles in water. This procedure has many advantages including operational simplicity, excellent yields, short reaction times, low cost, and green conditions.

Introduction

Green chemistry is the design of chemical products and processes that minimize or eliminate the use of hazardous reagents or solvents and offers valuable synthesis of expected products in an economically manner. With the successful synthesis of green nanoparticles (NPs), recently numerous methodologies are designed to synthesize metal NPs employing biological methods where enzymes,[1] microorganisms,[2] and plant extracts [3-5] play an important role in the formation of NPs. Among biological methods, using plants for NPs synthesis is safe, inexpensive, and the best eco-friendly alternative to more complex chemical and physical synthetic procedures so can be used as an economic and suitable substitute for the large scale production of metal NPs.[6]

Over the past few decades, nanoparticles of noble metals have attracted much attention from scientists due to significantly physical, chemical, and biological properties. Silver NPs play an important and key role in the field of the researchers due to possess tremendous applications in the areas such as drug delivery,[7] imaging,[8] anti-viral agents in the health industry,[9] textile coatings, and a number of environmental applications.[10] Also, these NPs have been used extensively as catalyst in chemical reactions.[11-13]

Multicomponent reactions (MCRs) are the best tool in organic synthesis and medicinal chemistry as they permit rapid access in generating molecular complexity and variation structures *via* a single synthetic operation.[14,15] The significant features of MCRs include rapidity, diversity, efficiency, atom-economy, and environmental amiability.[16] Because of these remarkable advantages, design and improvement of new MCRs with environmentally benign protocols have been recognized as one of the key topics in synthetic chemistry.

Now days, water has emerged as a desired solvent in organic synthesis because it is available, non-toxic, non-corrosive, non-flammable, cheap, and environment friendly. Additionally, reactions in water illustrate unique reactivity and selectivity. The use of water as green solvent in organic chemistry was rediscovered in the 1980s in Breslow's work, which was highlighted that hydrophobic effects could strongly enhance the rate of some organic reactions. In this effect, hydrophobic molecules in aqueous solution are surrounded by a cage of water molecules. The condensation of individual molecules to form local domains consisting of multiple hydrophobes surrounded by water cages is favored by the minimization of the surface area of contact between the hydrophobic and hydrophilic domains. Therefore, the acceleration of a reaction through the hydrophobic effect is expected to correlate with the hydrophobic surface area of the reactants. [17] Accordingly, the development of synthetically useful reactions in water is gaining considerable attention of the scientific society.[18-21]Spirooxindole cores are the main skeleton for various alkaloids and pharmacologically important compounds[22] with broad range of useful pharmacological properties and biological activities such as; antimicrobial,[23] antitumor,[24] antibiotic agents,[25] and microtubule assembly inhibitors,[26] for example spirotryprostatin A, ptropodind and alstonisine (Fig. 1), so considerable efforts have been devoted to find efficient and simple synthetic methods to prepare compounds containing spirooxindole nucleus.[27]

Notes and references

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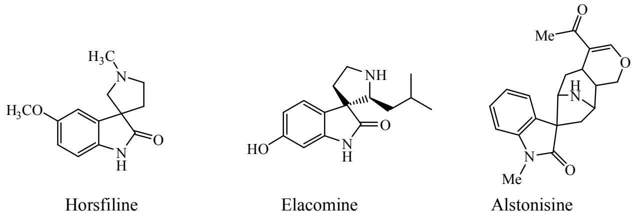


Fig. 1 Selected spirooxindole natural products

In the present study, we demonstrate a facile, efficient, and green biological approach to fabricate Ag NPs using aqueous extract of *Ferula Latisecta* leaves extract, then in continuing our efforts towards the development of efficient and environmentally benign methods for the synthesis of spirooxindoles,[28-30] synthesized Ag NPs were used as powerful and highly efficient catalyst in one-pot preparation of spirooxindole derivatives in water.

Experimental

The chemicals used in this work were obtained from Fluka and Merck and were used without purification. Melting points were measured on an Electrothermal 9200 apparatus. IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded in DMSO–d_6 solvents on a Bruker DRX-400 spectrometer. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. Nanostructures were characterized using a Holland Philips Xpert X-ray powder diffraction (XRD) diffractometer (Cu K, radiation, k = 0.154056 nm), at a scanning speed of 2°/min from 10° to 100°/(2θ). Transmission electron microscopic (TEM) was performed with a Jeol JEM- 2100UHR, operated at 200 kV that were previously air-dried. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company). *Ferula Latisecta* was collected from Hezar masjed mountain-Khorasan-Iran.

a) Preparation of plant aqueous extracts

Aqueous extract was prepared by mixing 5 g of dried fresh leaves powder of *Ferula Latisecta* with 100 mL of water with constant stirring on a magnetic stirrer. The mixture was boiled for 15 min before being decanted, and was cooled and filtered through Whatman No. 1 filter paper. The boiled extract was refrigerated and used for further experimental procedures.

b) Synthesis of AgNPs

Silver nanoparticles were prepared by adding 0.5 ml of *Ferula Latisecta* leaves extract to 20 ml of 1 mM aqueous silver nitrate solution. The reaction mixture containing silver nitrate solution and plant extract was agitated and incubated at 70 °C, 500 rpm for 48 h. The solution turned from yellowish to dark brown. The obtained nanoparticles solution was purified by repeated centrifugation at 12,000 rpm for 20 min followed decanted and dried at 60 °C in oven over night.

c) Synthesis of 2-Amino-3*H*-spiro[furo[3,4-*g*]pyrido[2,3-*d*]pyrimidine-5,3'-indoline]-2',4,6(8*H*,9*H*) -trione in the presence of Ag NPs (4a)

A mixture of isatin **1a**, tetronic acid **3a**, 2, 6-diaminopyrimidin-4(3*H*)-one **2c,** and Ag NPs (2 mol%) in water (5 ml) was stirred at reflux conditions for an appropriate time. After completion of the reaction as indicated by TLC, the solution was cooled to room temperature, then the precipitated product was filtered and washed with water (10 mL), dried to afford the crude product. The crude precipitate dissolved in DMF and centrifuged to separate the catalyst and latter the catalyst was washed several times with EtOH, then dried and reutilized four times for the same reaction. After separation of catalyst, 10 ml water was poured in the mixture of reaction to precipitate of the product. Then, the precipitate was filtered and washed with water and recrystallized by EtOH to afford the pure product white powder (Yield: 87%). mp: >350ºC. IR (KBr) (ν_{max}/ cm^{-1}): 3420, 3156, 1751, 1713, 1638; ^{1}H NMR (DMSO-d_6, 400 MHz): δppm: 4.84 (2H, s, OCH$_2$), 6.58-7.06 (6H, m, ArH and NH$_2$), 10.06 (1H, s, NH), 10.19 (1H, s, NH), 10.47 (1H, s, NH); ^{13}C NMR (DMSO-d_6, 100 MHz): δ_{ppm}: 47.4, 65.7, 91.0, 98.7, 108.9, 121.5, 123.8, 127.9, 136.0, 142.7, 155.0, 155.4, 158.8, 161.2, 170.2, 179.0; MS: m/z 337.

Results and discussion

During the past few years, significant interest has been focused on the development of new protocols for environmentally benign processes that are both economically and technologically achievable. Several methods have been reported to synthesize of Ag NPs includes both chemical and biological approaches.[31,32] According to green chemistry principles, using bio-based matter in synthesizing materials is of great interest to the scientific community. In comparison with chemical approaches, bio-based methods were fast without using any toxic and hazardous chemicals and by-products.

In the present study, firstly, Ag NPs were successfully synthesized from silver nitrate solution as a metal salt precursor through a simple green route using the leaf extract of *Ferula Latisecta*. *Ferula Latisecta* leaf extract not only reduced Ag$^+$ ions to Ag NPs, but also acted as capping and stabilizing agent. It was observed that the color of the solution turned dark brown color within appropriate time, which indicated the formation of silver nanoparticles (Fig. 2). Synthesized nanoparticles were characterized using UV–Vis spectroscopy, powder X-ray diffraction (XRD), and transmission electron microscopy (TEM).

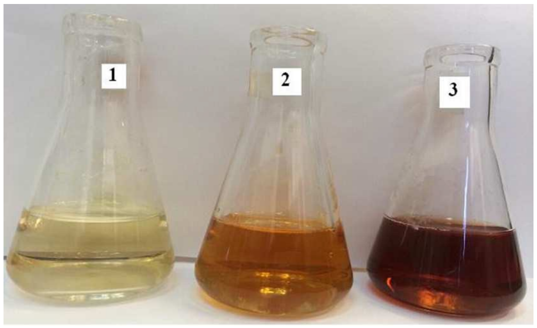


Fig. 2 Aqueous solution of 1 mM AgNO$_3$ with *F. Latisecta* leaf extract. After 8 h incubation (1), after 20 h incubation (2) after 40 h incubation (3)

Characterization of silver nanoparticles

UV-Vis spectrophotometer analysis

The UV–Vis spectra showed maximum absorbance peak at 420 nm, which increased with time of incubation of silver nitrate with the plant extract (Fig 3). The curves show increased absorbance in various time intervals (8 h, 20 h, and 40 h) and the peaks were noticed at 420 nm corresponding to the surface plasmon resonance of silver nanoparticles. The observation indicated that the reduction of the Ag$^+$ ions took place. It is reported earlier that absorbance at around 430 nm for silver is a characteristic of these nobel metal particles.[33]

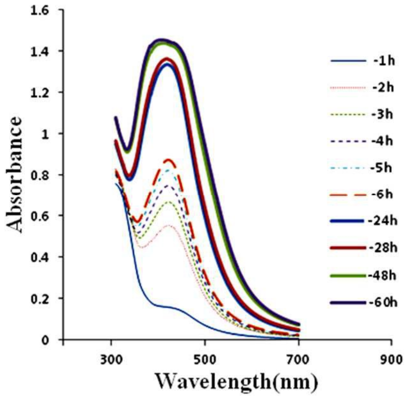


Fig. 3 UV–Visible spectra of aqueous 1 mM silver nitrate with *F. Latisecta* leaf extract at different time intervals in 70 °C with 0.5/20 concentration.

X-ray diffraction (XRD)

The X-ray diffraction (XRD) has proven to be a valuable research tool to prove the formation of AgNPs. The X-ray diffraction pattern of Ag NPs produced by leaf extract is shown in Fig. 4. The XRD pattern shows four characteristic peaks in the whole spectrum. The peaks at 2Θ values of 38.68°, 44.1°, 64.11°, and 77.4° corresponding to 111, 200, 220, and 311 crystalline planes of face centered cubic (fcc) crystalline structure of metallic silver which was matched with the database of Joint Committee on Powder Diffraction Standards (JCPDS) file No. 04–0783. The mean size of Ag NPs was also calculated using the Debye–Scherrer's equation it shows that diameter of produced Ag NPs is about 20 nm.

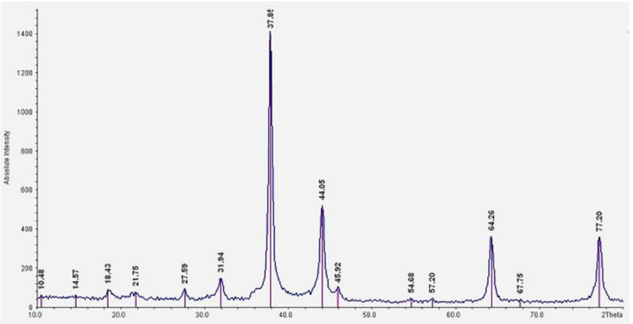


Fig. 4 The X-ray diffraction patterns of biosynthesized Silver nanoparticles.

Transmission Electron Microscopy (TEM)

Morphology and particle size of Ag NPs were characterized using TEM technique. As can be seen in Fig. 5 the morphology of nanoparticles is spherical without aggregation of particles. Additionally the TEM image shows that the AgNPs nanoparticles have a mean diameter of about 20–30 nm.

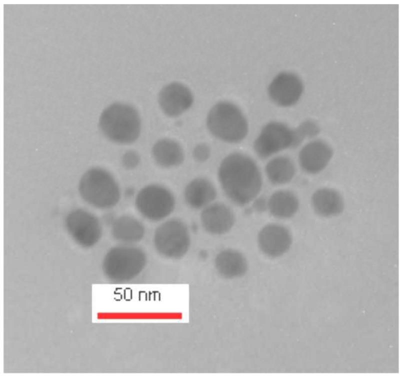
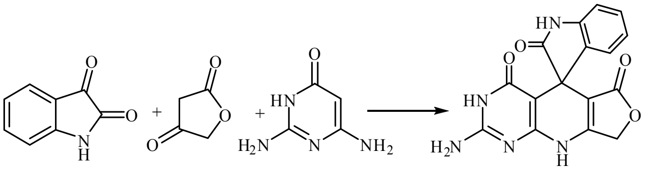


Fig. 5 The TEM image of biosynthesized silver nanoparticles

After characterizations, in order to study the efficiency of Ag NPs, we examined its catalytic activity in a one-pot, three component condensation reaction of isatin **1a**, tetronic acid **3a**, and 2,6-diaminopyrimidin-4(3*H*)-one **2c** as a model reaction for the synthesis of 2-Amino-3*H*-spiro[furo[3,4-*g*]pyrido[2,3-*d*]pyrimidine-5,3'-indoline]-2',4,6(8*H*,9*H*) -trione (**4a**) (Scheme 1). Detailed literatures survey toward the synthesis of these compounds revealed that only two papers have been published.[34] Regarding the importance of these compounds because of their biological and medicinal properties, we have tried to access these compounds in short reaction time, excellent product yields, and green way.



1a **3a** **2a** **4a**

Scheme 1. Model reaction for the synthesis of **4a.**

In our initial investigations, the effect of solvent was evaluated in the model reaction in the presence of Ag NPs (2 mol %) as catalyst. The results are summarized in Table 1. As can be seen in this table, water provided higher yields than those using other organic solvents under reflux conditions and no product was formed when the reaction was carried out in non-polar solvents, such as toluene and n-hexane (Table1, Entries 6 and 7).

Table 1 Screening of solvents for the synthesis of **4a**[a].

Entry	Solvent	Time (min)	Yield (%)[b]
1	H_2O	15	87
2	EtOH	60	64
3	CH_2Cl_2	120	-
4	THF	120	-
5	CH_3CN	120	<50
6	Toluene	120	-
7	n-Hexane	120	-

[a]Reaction condition: isatin (1 mmol), tetronic acid (1 mmol), 2, 6-diaminopyrimidin-4(3*H*)-one (1 mmol), solvent (5 mL) Ag NPs (2 mol%) under reflux condition.

[b]Isolated yields of the pure prouduct.

In the next step, in order to show the advantage of Ag NPs over other catalysts, the model reaction was performed in the presence of different catalysts in refluxing water. The employed catalysts are listed in Table 2. As can be seen, the Ag NPs gives better yield in shorter reaction time. Additionally the reaction was tested without catalyst under heating conditions. It was found that no product was formed in the absence of catalyst even after prolonged reaction time (24 h).

Table 2 Comparison of various catalysts in the synthesis of **4a**[a]

Entry	Catalyst	Time (min)	Yield (%)[b]
1	Ag NPs	15	87
2	Ag Bulk	60	-
2	Au NPs	60	54
3	Nano MgO	60	>50
4	Nano ZnO	60	>50
5	Nano TiO_2	60	>50
6	Nano $MnFe_2O_4$	360	82[34]
7	-	24 h	-

[a] Reaction condition: isatin (1 mmol), tetronic acid (1 mmol), 2, 6-diaminopyrimidin-4(3*H*)-one (1 mmol), catalyst (2 mol%), H_2O (5 mL).

[b] Isolated yields of the pure prouduct.

We further turned to testing the effect of the amount of the catalyst with values of 1, 2, 5, and 10 mol% of Ag NPs in model reaction respectively. It was observed that 2 mg loading of the catalyst provides the maximum yield in minimum time (Table 3, Entry 2). The results indicated that 2 mol% of catalyst was optimal. Higher amounts of catalyst did not lead to a significant change in yield and time (Table 3).

Table 3 Effect of the amount of the AgNPs as a catalyst in the model reaction[a]

Entry	Catalyst (mg)	Time (min)	Yield (%)[b]
1	1	15	65
2	2	15	87
3	5	15	88
4	10	15	88

[a] Reaction condition: isatin (1 mmol), tetronic acid (1 mmol), 2, 6-diaminopyrimidin-4(3*H*)-one (1 mmol), H_2O (5 mL), under reflux condition.

[b] Isolated yields of the pure prouduct.

The recovery of catalyst is most important in green synthetic process. Finally, we examined the recyclability of the Ag NPs for four times with almost same catalytic activity as illustrated in Fig. 5, by using the reaction of isatin **1a**, tetronic acid **3a**, and 2,6-diaminopyrimidin-4(3*H*)-one **2c**, as model substrates under optimized conditions. Ag NPs were separated from the reaction mixture by centrifugation after addition of DMF as mentioned in the experimental section and washed with EtOH to remove the residual product. After being dried, it was subjected to another reaction. The procedure was repeated for four cycles. The observed fact that yields of the product remained similar with no detectable loss. These experiments (Fig. 6), established the recyclability and reusability of the catalyst without significant loss of activity. The TEM analysis of the recovered Ag NPs revealed that the morphology and size of the nano particles remains relatively unchanged (Fig. 7), and a small aggregate was observed on the surface of the recovered Ag NPs.

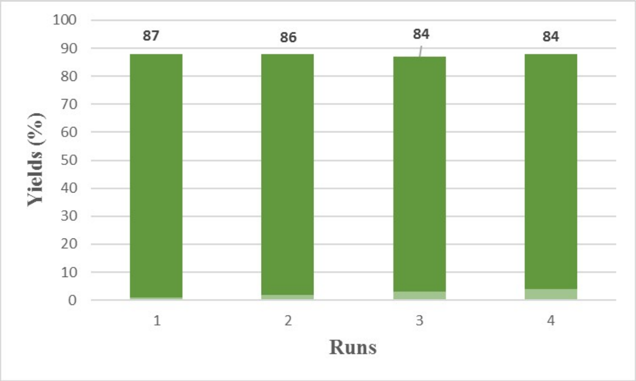


Fig. 6 Catalyst recyclability study on the synthesis of **4a.**

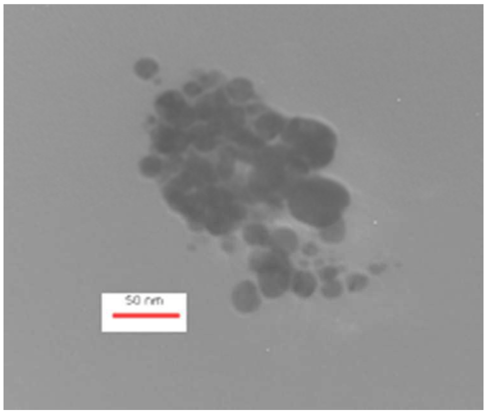
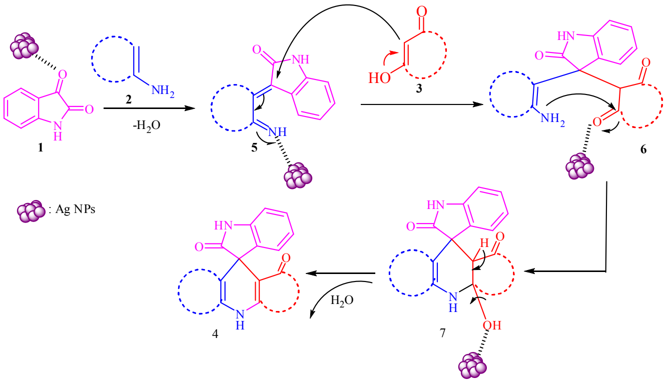


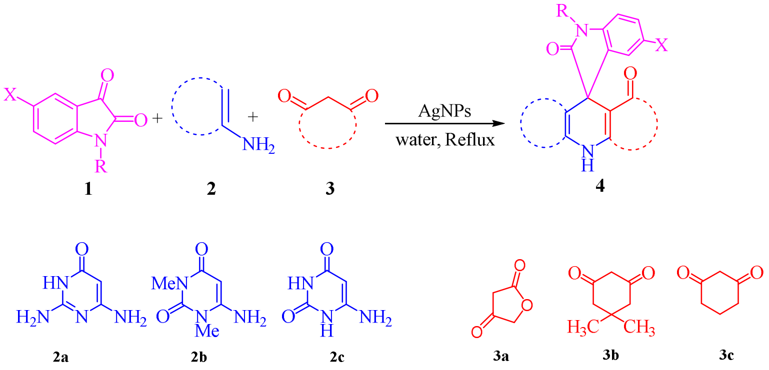
Fig. 7 TEM images of recovered Ag NPs as catalyst**.**

Although the exact mechanism for this three-component reaction has not been established at present, a plausible mechanism could be explained by the reaction sequence in Scheme 2. This mechanism is proposed on the basis of the analysis of similar multicomponent reaction of istain.[35] Reaction presumably proceeds *via* Knoevenagel condensation between isatin **1** as a highly active ketonic carbonyl and enamine **2** to give *α, β*-unsaturated compound **5**. Then, compound **5** converts to intermediate **6** through a Michael addition of *β*-diketone **3**. Finally, product **4** was obtained by an intramolecular cyclization process from compound **6** and followed by loss of 1 equiv of H_2O from compound **7**. Ag NPs as Lewis acid plays an important role in increasing the electrophilicity of the starting isatin and stabilizing the intermediates by the coordination of oxygen and nitrogen lone electron pairs with Ag NPs as shown is Schem 2.

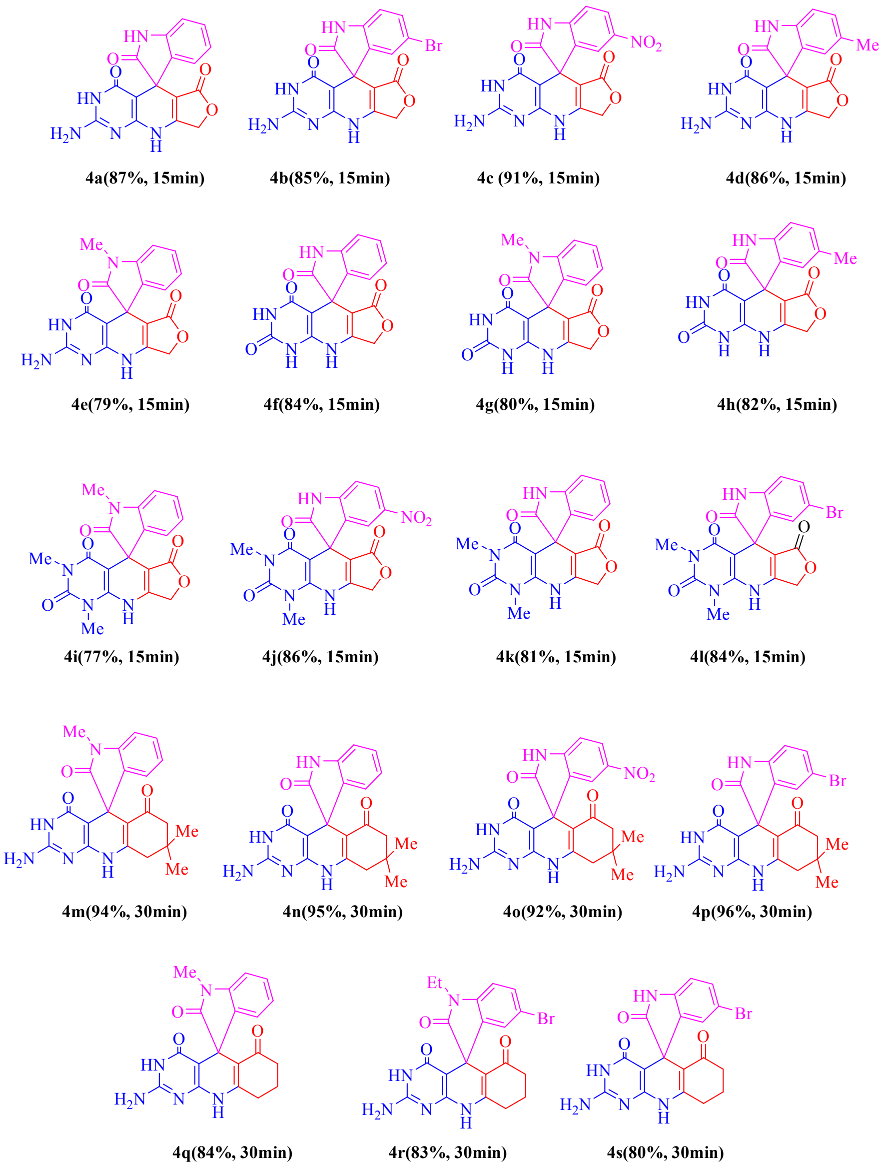
To explore the scope and limitations of this reaction, the optimized reaction conditions (water solvent, Ag NPs catalyst, 90 °C) were used for the construction of a library of spirooxindole derivatives from two CH acids tetronic acid and dimedone, isatins and 2, 6-diaminopyrimidine-4(3*H*)-one and uraciles as enamines (Scheme 3). All products were obtained in high to excellent yields, high purity, and short reaction times (Scheme 4).



Scheme 2. Proposed mechanism for the formation of 4.



Scheme 3. Preparation of spirooxindole derivatives in the presence of AgNPs.



4a(87%, 15min) 4b(85%, 15min) 4c (91%, 15min) 4d(86%, 15min)

4e(79%, 15min) 4f(84%, 15min) 4g(80%, 15min) 4h(82%, 15min)

4i(77%, 15min) 4j(86%, 15min) 4k(81%, 15min) 4l(84%, 15min)

4m(94%, 30min) 4n(95%, 30min) 4o(92%, 30min) 4p(96%, 30min)

4q(84%, 30min) 4r(83%, 30min) 4s(80%, 30min)

Scheme 4. Structure of produced spirooxindoles and yields.

Conclusions

In summary, we have developed a simple, inexpensive, eco-friendly, and green method for the synthesis of Ag NPs by the direct interaction of silver nitrate with *Ferula Latisecta* leaves extract as both reducing and capping agent in aqueous media. The application of Ag NPs as catalyst has been demonstrated for the synthesis of spirooxindole derivatives through the condensation reaction of isatins, two kinds of CH acids, and three kinds of enamines. The desired products were obtained in high to excellent yields, short reaction times, and high purities.

Acknowledgements

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Graphical Abstract

Green synthesis and characterization of silver nanoparticles using *Ferula Latisecta* leaf extract and its application as catalyst for the safe and simple one-pot preparation of spirooxindoles in water

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