

# Solvent-free coupling of aldehyde, alkyne, and amine over a versatile catalyst: Ag-functionalized mesoporous S, P-doped g- $C_3N_4$

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#### Abstract

In the present study, we introduce mesoporous  $g-C_3N_4/Ag$  co-doped with P and S which was designed and acquired by using mesoporous silica (SBA-15) as a hard templating agent and thiourea and chitosan phosphate as the dopants. The prepared catalyst was completely identified by FT-IR (Fourier-transform infrared spectroscopy), XRD (X-ray powder diffraction), FE-SEM (field-emission scanning electron microscopy), EDX (energy-dispersive spectroscopy), TEM (transmission electron microscopy), and Raman spectroscopy. The efficiency of the synthesized catalyst was evaluated toward the three-component coupling reaction, frequently named as A3 coupling. The higher activity of the prepared catalyst is because of the synergistic effects of phosphorus and sulfur co-doped together with Ag deposition. The desired products were achieved by an environmentally safe catalyst under the optimized conditions in high yields and short reaction time ranges.

**Keywords** Porous materials  $\cdot$  Three-component reactions  $\cdot$  Propargylamines  $\cdot$  Ag NPs  $\cdot$  G-C<sub>3</sub>N<sub>4</sub> nanocatalyst

#### Introduction

During the last decades, MCRs (multicomponent reactions) have been extensively employed in target-oriented synthesis (TOS) to design and prepare a vast group of organic and medical materials [1–3]. These ideal and unchallenging reactions from the point of amiable chemistry can be run a via one-pot procedure. The A3 coupling reaction (coupling reaction of terminal alkynes-aldehydes-amines) is referred to as one of the most popular MCRs and leads to the efficient preparation of propargylamines and water (the only by-product) [4–12]. The propargylamine derivatives are known to

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be multipurpose building blocks to form many organic compounds such as nitrogencontaining heterocycles and pharmaceutical products [13–16]. Alzheimer's and Parkinson's disorders can be treated by some of these medical active materials as well [17–21].

The 2D, stable and layered polymer, graphitic carbon nitride  $(g-C_3N_4)$  with the bandgap of 2.7 eV has been extensively employed in many research scopes including photocatalysis and catalysis [22]. G-C<sub>3</sub>N<sub>4</sub> as an efficient catalyst has been used for A3 coupling reaction [23], crossed-aldol condensation [24], Friedel–crafts reaction [25], Knoevenagel condensation [26], C–C couplings [27], oxidation reaction [28], Claisen–Schmidt condensation [29], reduction reaction [30], and so on. Commonly, the g-C<sub>3</sub>N<sub>4</sub> can be prepared through the calcination of nitrogen-containing precursors such as melamine [31], urea [32], thiourea [33], and cyanamide [34]. Unfortunately, despite features like non-toxic nature, low price, and effortless preparation method [35], the bulk g-C<sub>3</sub>N<sub>4</sub> suffers from the low surface area. So far, numerous papers have been published modifying the pristine g-C<sub>3</sub>N<sub>4</sub> and introducing porosity into the structure to obtain more active sites [36–38]. Its nitrogen-rich content as well as polymeric character enables the texture to be doped with foreign atoms and this would affect and enhance the catalytic activity.

Over the years, different transition metal compounds based on copper [39], nickel [40], iron [41], cobalt [42], silver [43], gold [44], mercury [45], and even copper/ruthenium salts [46] were implemented to catalyze the C–C and C–N coupling reaction. However, previous literature validated the coinage metals including copper, silver, and gold are the most intriguing transition metals to catalyze A3 coupling [47–49]. These metals may potentially boost the production of the target molecule through coordinating to the terminal alkyne and creating  $\pi$  complexes.

In connection with the points previously mentioned and based on our group's goal in modifying  $g-C_3N_4$ , and designing new catalysts [50–55], we report the design of novel mesoporous S, P-doped  $g-C_3N_4$ /Ag catalyst by using Pluronic P123 surfactant. To dope into the structure of the mesoporous  $g-C_3N_4$ , economical, natural, and accessible materials, thiourea, and chitosan phosphate were utilized. The prepared catalyst possesses a high surface area and exhibits acceptable catalytic performance for the A3 coupling reaction. The preparation of the recoverable catalyst was supported by IR, XRD, FE-SEM, EDX, TEM, and RAMAN analytical techniques.

#### Experimental

#### Materials

All the chemical reagents and solvents were used as acquired from Merck and Sigma-Aldrich companies with the highest analytical grade.

#### **Characterization techniques**

FT-IR (Fourier transformed infrared) spectra were determined on the Shimadzu spectrophotometer (8400 s) in the 400–4000 cm<sup>-1</sup> range. The powder XRD (crystalline structure) of the designed catalyst was carried out on Philips (PW1730) using Cu K<sub> $\alpha$ </sub> source of irradiation (wavelength of 1.54 Å). Scanning electron microscopy (FE-SEM) images and elemental investigation (EDAX) were employed by the FE-SEM TESCAN MIRA3 instrument. Transmission electron microscopy (TEM) micrographs were taken on TEM Philips (EM 208 s) microscope. The Raman spectra were conducted using TESCAN Raman Analyzer with a neodymium-doped yttrium aluminum garnet laser, which produces a wavelength of 532 nm.

### Synthesis of the mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst

#### Synthesis of the SBA-15 template (mesoporous silica)

Four grams of P123 copolymer surfactant was dissolved in 30 mL purified water and 80.5 mL HCl solution (2 M). After adding 8.5 g of tetraethyl orthosilicate (TEOS) in a dropwise manner to the above solution, the mixture was stirred at 40 °C for 8 h. Shortly after that, the mixture was transferred into an autoclave and heated to 100 °C. After 20 h, the autoclave was cooled to the room temperature, and the resultant product was obtained by filtering, washing, and eventually drying at 65 °C in the oven [56].

#### Synthesis of chitosan phosphate

Two grams of chitosan, 10 g of urea, and 2 g of orthophosphoric acid were added to the round-bottom flask containing 20 mL of DMF solvent. The mixture was heated to 150 °C for 1 h. After cooling, the precipitate was centrifuged, washed with ultrapure water several times, and freeze-dried [57].

#### Synthesis of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>

A mixture of SBA-15 (1 g), chitosan phosphate (2 g), thiourea (4 g), and deionized water (40 mL) was heated for almost 12 h. After removing the solvent under reduced pressure, the obtained sample was dried in the oven during the night. The grounded solid was transferred into the crucible and calcined at 500 °C for 2 h with a heating rate of 20 °C/min. Finally, to leach out the silica template NaOH solution (2 M, 1:1 EtOH:  $H_2O$ ) was added to the calcined sample from the previous step at 80 °C and stirred for 4 h. As the last step, the mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub> was obtained by filtering, washing, and drying the resultant material at 80 °C.

#### Synthesis of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag

To load Ag NPs on the surface of graphitic support, 470 mg of mesoporous S, P-doped  $g-C_3N_4$  was added to DI water (100 mL) and sonicated. Afterward,

4.72 mL of  $AgNO_3$  solution (10 mg mL<sup>-1</sup>) was added to the reaction mixture. As the last step, 15 mL of the reducing agent (NaBH<sub>4</sub>) (210 mg) was injected into the suspension under mechanical stirring. After 12 h, the synthesized catalyst was isolated through the centrifuge, washed with H<sub>2</sub>O, and dried at 80 °C.

# The common procedure for A3 coupling reaction catalyzed by mesoporous S, P-doped $g-C_3N_4/Ag$

As a model method, 0.02 g of mesoporous S, P-doped g- $C_3N_4/Ag$  catalyst was added to the RB-flask containing 1 mmol of aromatic aldehyde, 1.2 mmol of piperidine, and 1.5 mmol of terminal alkyne. The reaction mixture was stirred at 100 °C and under solvent-free condition. The progress of the coupling reaction was followed by TLC (thin layer chromatography) until the complete conversion of substrates to propargylamine derivatives. Thereafter, the catalyst was removed by centrifuge and washed with ethanol. The pure target compounds were obtained using silica gel column chromatography (ethyl acetate/hexane 4:6). Eventually, the reaction products were recognized by the gas chromatography (GC) technique. <sup>1</sup>HNMR spectra of some selected compounds are given in SI.

Spectral data of *N*-(1,3-diphenyl-2-propynyl) piperidine: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ(ppm): 7.28–7.67 (m, 10H), 4.81 (s, 1H), 2.59 (s, 4H), 1.58–1.65 (m, 4H), 1.45–1.49 (m, 2H).

## **Results and discussion**

#### **Catalyst fabrication**

The synthetic path for preparing the mesoporous S, P-doped g- $C_3N_4/Ag$  was mentioned in the previous section. In brief, after synthesis of mesoporous silica and chitosan phosphate they were reacted with thiourea to form mesoporous S, P-doped g- $C_3N_4$ . As a final step, Ag NPs were immobilized on the surface of the prepared mesoporous S, P-doped g- $C_3N_4$  (Scheme 1).

#### **Catalyst characterization**

To underline the existence of corresponding functional groups, FT-IR investigation was done. Figure 1 depicts Fourier-transform infrared spectra of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub> and mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag samples. The appeared peaks in the spectrum of both samples are following the reported literature for mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub> [58]. The observed bands at the range of 1200–1600 cm<sup>-1</sup> are related to the CN stretching modes of g-C<sub>3</sub>N<sub>4</sub>. The broad peak centered at 3350 cm<sup>-1</sup> refers to the free NH groups of the graphitic structure as well as hydroxyl vibration of adsorbed water. After co-doping of P and S atoms, all the characteristic peaks in connection with g-C<sub>3</sub>N<sub>4</sub> could also be observed in the mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag, proving that the original graphitic CN network



Scheme 1 General route for the synthesis of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst

was maintained intact. Obviously, the peak intensity for mesoporous S, P-doped  $g-C_3N_4/Ag$  sample was decreased due to the successful assembly of Ag NPs on the surface of the graphitic support.

In this study, XRD analysis was surveyed to confirm the phase purity and crystalline texture of the samples. The X-ray diffraction patterns corresponding to the mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>, and mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag samples are demonstrated in Fig. 2. Two diagnostic peaks at 13.1° and 27.3° (100 and 002 planes) are recognized for graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) based on the JCPDS card No. 87–1526 [59]. The peaks of S, P-doped CN show positions similar to those of g-C<sub>3</sub>N<sub>4</sub> but rather broadened, suggesting that more defects in the structure appear



Fig.1 FT-IR spectra of mesoporous S, P-doped g- $C_3N_4$  sample and mesoporous S, P-doped g- $C_3N_4$ /Ag catalyst

along with phosphorus and sulfur doping [60]. Moreover, the peak location of the (002) plane is shifted from 27.3° to 26.45° (mesoporous S, P-doped g- $C_3N_4$ ) and 26.95° (mesoporous S, P-doped g- $C_3N_4/Ag$ ), which is because of the heteroatoms (phosphorus and sulfur) doping into the lattices of g- $C_3N_4$ , leading to greater lattice



Fig. 2 XRD patterns of the mesoporous S, P-doped g- $C_3N_4$  and mesoporous S, P-doped g- $C_3N_4/Ag$  samples

distortions and interplanar distance [61]. XRD patterns indicated that the S, P codoping has no significant influence on the original crystal structure of  $g-C_3N_4$ . No evidence for any impurity was detected. After immobilization of Ag NPs, four distinct peaks (38.12° (111), 44.30° (200), 64.40° (220), and 77.26° (311)) were noticed which were assigned to the cubic structure of silver, Ag (0) based on the ICCD card No. 04–0783 [62]. Furthermore, the average crystalline size of the Ag NPs in their zero oxidation state was calculated to be 25 nm (based on the Scherrer's equation  $D = k\lambda/\beta cos\theta$ ).

FE-SEM as well as mapping analysis was carried out to reveal the surface morphology and syntax of the prepared mesoporous S, P-doped  $g-C_3N_4/Ag$  sample (Fig. 3). As can be realized from Fig. 3 the porous nature of the synthesized mesoporous S, P-doped  $g-C_3N_4$  did not alter after Ag nanoparticles sedimentation. Figure 3a–e exhibits the uniform distribution of C, N, P, S, and Ag elements over the surface of the  $g-C_3N_4$  support. Besides, successful immobilizing of these NPs on the surface of the  $g-C_3N_4$  support can be verified by TEM images (Fig. 4).

To pursue the chemical composition of the prepared samples (chitosan phosphate and mesoporous S, P-doped g- $C_3N_4$ , and mesoporous S, P-doped g- $C_3N_4/Ag$ ), EDS analysis was performed. The illustrated spectrum in Fig. 5 represents corresponding peaks to C, N, O, and P elements, which is related to chitosan phosphate. For the mesoporous S, P-doped g- $C_3N_4$  sample, the presence of C, N, partially S (8.11 W%) and P (1.43 W%) peaks in the spectrum declares the satisfactory preparation of the graphitic support (Fig. 6). For the mesoporous S, P-doped g- $C_3N_4/Ag$  catalyst, the presence of Ag (7.2 W%) peaks in the spectrum declares the convincing immobilization of silver NPs on the graphitic support (Fig. 7).

To study the vibrational feature of carbon-containing material, the Raman spectroscopy was conducted. The Raman spectra of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst is exhibited in Fig. 8 and in the range of 1000–2000 cm<sup>-1</sup>. The typical D and G bands for the mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst is positioned at nearly 1361 and 1536 cm<sup>-1</sup>, respectively. The Raman spectra of excited doped carbon-containing materials show two characteristic resonances (D and G bands) located at about 1320 and 1590 cm<sup>-1</sup> [63]. The in-plane stretching modes of carbon (G-band) are related to graphene structure, while the out of plane modes of phonon (D band) refer to the amorphous carbon structures [64]. Also, the I<sub>G</sub>/I<sub>D</sub> intensity ratio is calculated to be 1.13, proving the coordination of metal nanoparticles (Ag) to the  $\pi$  electrons of the graphitic support [65].

## Evaluating the efficiency of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst in the A3 coupling reaction

Once the structure of the designed catalyst has been verified, the catalytic ability of mesoporous S, P-doped  $g-C_3N_4/Ag$  was determined in the A3 (three-component) coupling reaction of aromatic aldehydes, secondary amines, and terminal alkynes. Benzaldehyde, piperidine, and phenylacetylene were chosen as substrates to establish the title reaction.



Fig.3 FE-SEM images of mesoporous S, P-doped  $g-C_3N_4/Ag$  catalyst with different magnifications and EDS-mapping pictures of the catalyst a-e

Diverse factors such as catalyst amount, temperature, and solvent were tested and the summarized outcomes are given in Table 1. Originally, the effect of catalyst loading was examined and due to Table 1, the expected product was achieved in the presence of 20 mg of catalyst. The model reaction did not proceed in the absence of the catalyst even after 10 h. This matter emphasizes the mandatory rule of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst in the reaction progress (Table 1, entry 1, 2, 3). To explore the influence of the solvent's nature on this conversion, different solvents including protic (H<sub>2</sub>O, EtOH) and aprotic ones (Toluene, THF) were applied to run the model test. From the entry 3, it is clear that under the solvent-free condition the generation yield of the product is ideal. Eventually, to investigate the impact of temperature, the A3 coupling reaction was managed with 20 mg of catalyst and under the solvent-free condition at dissimilar temperatures. 100 °C was chosen as the superior temperature to accomplish the



Fig. 4 TEM image of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst



Fig. 5 EDS analysis of chitosan phosphate

conversion. Entry 3 mentions the optimized condition for the production of propargylamine derivatives.

Following optimizing the reaction condition, the addition of terminal alkynes to secondary-type amines and aromatic aldehydes to form propargylamine derivatives in a solvent-free condition by using a silver-based catalyst has also been investigated. The summarized results in Table 2 reveal the ability of the prepared catalyst



Fig. 6 EDS analysis of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub> support



Fig. 7 EDS analysis of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst



Fig. 8 Raman spectra of mesoporous S, P-doped g-C<sub>3</sub>N<sub>4</sub>/Ag catalyst

Entry	Catalyst (mg)	T (°C)	Solvent	Time (min)	Con. %
1	_	100	Solvent-free	10 h	trace
2	$m-g-C_{3}N_{4}(10)$	100	Solvent-free	10 h	trace
3	m-S, P-doped $g-C_3N_4(10)$	100	Solvent-free	10 h	trace
4	10	100	Solvent-free	120	80
5	20	100	Solvent-free	75	97
6	30	100	Solvent-free	120	95
7	20	reflux	toluene	120	20
8	20	reflux	THF	120	trace
9	20	reflux	EtOH	120	10
10	20	reflux	H <sub>2</sub> O	120	trace
11	20	r. t	Solvent-free	120	trace
12	20	80	Solvent-free	120	85
13	20	130	Solvent-free	120	95

 Table 1 Optimization conditions for three-component coupling reaction of benzaldehyde, piperidine, and phenylacetylene

to be applied to aromatic aldehydes with different functional-groups (electron-rich and electron-withdrawing) in diverse ring-positions. The substrates converted to the desired product and water (the only green by-product) in excellent yields and acceptable time ranges between 75 and 90 min.

The effectiveness of mesoporous S, P-doped  $g-C_3N_4/Ag$  catalyst was compared with other published strategies for the Benzaldehyde/ Piperidine/ Phenylacetylene

Entry	Aldehyde	Amine	Alkyne	Product	Time	Yield
1	СНО	O N H		Ph	75	97
2	СІСНО			Ph Cl	85	95
3	СНО	O N H		Ph Ph	90	89
4	Br			Ph Br	80	94
5	МеО			Ph OMe	80	89
6	СНО	O N H		Ph HO	80	87
7	СНО		C	Ph	90	89
8	СНО			Ph Cl	75	95
9	СНО	N H		Ph	75	97

 
 Table 2 Coupling reaction of aromatic aldehyde, terminal alkyne, and amine catalyzed by the silverbased catalyst





Condition: Benzaldehyde (1 mmol), Amine (1.2 mmol), phenylacetylene (1.5 mmol), 0.02 g of catalyst, and no solvent. The isolated yield is based on the conversion or completion of the reaction in given time.

coupling reaction (Table 3). According to the data in Table 3, our prepared green and unchallenging catalyst showed much more catalytic efficiency.

Based on the reported mechanistic studies in past literature [75], we propose a tentative mechanism for the generation of propargylamines in the presence of the mesoporous S, P-doped g- $C_3N_4/Ag$  catalyst (Scheme 2). The feasible mechanism for the coupling reaction over Ag nanoparticles is suggested that involves the C–H bond of alkyne activation. After in situ generations of iminium ion from the initial amine

Entry	Catalyst	Condition	Time (h)/Yield	Refer- ences
1	Silver-graphene nanocomposite	CH <sub>2</sub> Cl <sub>2</sub> , 60 °C	24/86	[66]
2	ZnO-IL/Ag NPs	H <sub>2</sub> O, reflux	3/92	[ <mark>67</mark> ]
3	Ag <sub>3</sub> PW <sub>12</sub> O <sub>40</sub> (AgTPA)	Acetonitrile, 80 °C	5/92	[68]
4	AgY zeolite	100 °C	15/81	[ <mark>69</mark> ]
5	CuO/GNS	Acetonitrile, 82 °C	5/89	[70]
6	Ag/EOs orange NPs	Toluene, 80 °C	8/96	[71]
7	Ag-CIN-1	H <sub>2</sub> O, 40 °C	3/98	[72]
8	$(Ag_xNi_{(1-x)})_yRGO_{(100-y)}$	Acetonitrile, 100 °C	12/88	[73]
9	Ag-Co/Hi-ZSM-5	H <sub>2</sub> O, 75 °C	15/84	[74]
10	Present study	Solvent-free, 100 °C	75 min/97	_

 Table 3
 Three-component coupling reaction catalyzed by different catalysts



Scheme 2 Tentative mechanism for the silver-catalyzed coupling reaction

and aldehyde, it reacted with the silver-acetylide intermediate to form the corresponding product (propargylamine) and reproduce the silver-based catalyst to complete the reaction.

The recyclability of the mesoporous S, P-doped g- $C_3N_4/Ag$  catalyst was studied for model reaction in seven consecutive runs (Fig. 9). The catalyst was regained through centrifuge by the completion of each cycle. The catalyst was then washed with ethanol, dried in the oven, and reused for another run. The catalyst can be easily recycled for seven tests with inconsiderable loss of activity. As illustrated in Fig. 10 the XRD patterns also affirms the recycling ability of the mesoporous S, P-doped g- $C_3N_4/Ag$  catalyst after 7 successive runs.



Fig. 9 Reusability test of the catalyst for the model reaction



Fig. 10 XRD patterns of the catalyst and the recycled one

#### Conclusion

In conclusion, an exceptionally cost-effective silver-catalyzed A3 (three-component) coupling reaction of aromatic aldehyde, terminal alkyne, and amine has been accomplished through C-H activation. The catalyst preparation, procedure, and work-up were plain, and no organic solvent or activator was needed. The catalyst managed a broad range of starting materials for the generation of the propargylamine derivatives with excellent yields. Recyclability of the catalyst was up to seven repetitive runs with no appreciable loss of activity. Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s11164-021-04453-3.

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#### Declaration

Conflicts of interest There are no conflicts to declare.

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