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Biosynthesis of CuO nanoparticles using aqueous extract of Ziziphus mauritiana L. leaves and their Catalytic performance for the 5-aryl-1,2,4-triazolidine-3-thione derivatives synthesis

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ABSTRACT

Copper oxide (CuO) nanoparticles (NPs) were synthesized by biological method using aqueous extract of *Ziziphus mauritiana* L. leaves as a reducing and stabilizing agent. The merits of this procedure are an easy operation, use of cheap, nontoxic and environmentally benign precursors. The structural and morphological properties of the as-prepared nanocatalyst were done by using XRD, SEM, EDX, TEM and BET-surface area. Furthermore, these NPs are used as an effective nanocatalyst for simple and one-pot synthesis of 5-aryl-1,2,4-triazolidine-3-thione derivatives. The reaction steps including imine formation, cyclization, condensation and aromatization occurs, without use of any oxidizing or reducing reagents. The present methodology offers several remarkable merits such as shorter reaction time, mild reaction conditions, excellent yield, simplicity, safer reaction pathway, easy workup and recyclable catalyst without any significant loss in catalytic activity and can be used for large scale synthesis. Hence, this study describing the synthesis of CuO NPs by effective biogenic method followed by the investigation of potent catalytic activities may be useful for nanochemistry research opening new horizons in this field.

Keywords : Nanotechnology, *Ziziphus mauritiana* L., CuONPs, Recyclable catalyst.

I. INTRODUCTION

Nowadays metal oxide NPs has got great attention in catalysis because of their redox properties, coordination environment of surface atom and oxidation state at surface layer. Extensive surface area of nanoparticles and number of active sites than the bulk material, makes NPs more practicable catalyst for organic synthesis.[1–5] High atom efficiency, moderate reaction conditions, simple separation of product and recyclability of the catalysts are the merits of NPs catalyzed reactions.

Prominent applications of CuO NPs generated a great deal of interest in biomedical[6], catalysis[7], lubricant[8] and gas sensing[9]. CuO NPs were synthesized using microwave irradiation, ionic liquid assisted, sol-gel technique, sonochemical, electrochemical and thermal treatment[10–15] are reported recently. The existing methods have some drawbacks due to use of some drastic conditions such as use of toxic chemicals, external additives required during the reaction, high temperature etc. Therefore, there is massive scope for the development of green protocol for the synthesis of CuO NPs. Nowadays, nanomaterial synthesis using plant extract is receiving lot of attention with some advantages over chemical methods.[16–18] The inspection of the literature exposed some plant extract mediated synthesis of CuO NPs by, *Camellia japonica*[19] *Rauvolfia serpentina*[20], *Leucaena*

leucocephala[21], *Calotropis gigantean*[22], *Aloe barbadensis*[23], *Ficus religiosa*[24], *Albizia lebbeck*[25] and *Acanthospermum hispidum*[26] have been reported.

The synthesis of 5-aryl-1,2,4-triazolidine-3-thione derivatives was carried out by using synthesized CuO NPs. Literature survey reveals that the sulfur containing 1, 2, 4-triazole have been reported, as antidepressant[27,28], anti-tubercular[29], antibacterial[30,31], anti-HIV[32], antimicrobial activity[33] and antifungal activity[33]. The biological applications of, 5-aryl-1,2,4-triazolidine-3-thione derivatives have fascinated attention of many researchers. Analysis of literature exposes that the 5-aryl-1,2,4-triazolidine-3-thiones involved the use of $[C_{16}MPy]AlCl_3Br$[34], [2-HMPyBSA]HSO_4[35], PEG-400[36], glycine nitrate[37], Sm_2O_3/FAp[38], Fe-FAp[39], sulfamic acid[40], DMAP[41], $[(Py)_2SO][HSO_4]_2$[42] for the synthesis.

The present study we report CuO NPs catalyzed one pot methodology that comprises both, condensation and cyclization of aldehyde with thiosemicarbazide. CuO NPs assisted synthesis improves the rate of reaction towards 5-aryl-1,2,4-triazolidine-3-thione.

II. METHODS AND MATERIAL

2.1 Characterization techniques

The morphology and elemental composition of the fabricated CuO NPs were examined by field emission scanning electron microscopy (FESEM, FEI, Nova Nano SEM 450), FESEM coupled energy-dispersive X-ray spectroscopy (EDX, Bruker, XFlash 6I30). Find the exact morphological structures and size of the CuO NPs using transmission electron microscopic (TEM) analysis is done by using a PHILIPS-CM200. The crystallinity and crystal phases were characterized by X-ray diffraction (XRD, Brukar, D8-Advanced Diffractometer) pattern measured with Cu- Kα Radiation (λ= 1.5406 Å) in the range of 5–90°. These all characterization results have been reported in our previous research work[43].

2.2 General procedure for the synthesis of 5-phenyl 1,2,4-triazolidine 3-thione derivative.

Equimolar mixture (10 mmol) of aldehyde and thiosemicarbazide were mixed with CuO NPs (5 mg) in ethanol (5 mL) taken in a round bottom flask. The reaction mixture was then heated to 70 ^{0}C for appropriate time. After completion of the reaction, the reaction mixture was diluted with the ethyl acetate and the catalyst was separated by centrifugation. The crude product was then purified by recrystallization to get pure product.

2.3 Spectral data of synthesized compounds

5-Phenyl -1,2,4-t riazol idine-3-thione (3a):
White solid, Mp 152-154 °C; 1H-NMR (500 MHz, DMSO-d6): δ 11.49 (s, 1H, NH), 8.22 (s, 1H, NH), 8.04 (s, 1H, CH), 8.0 (s, 1H, NH), 7.80 (d, 2H, ArH, J = 6.8 Hz), 7.39 (t ,3H, ArH, J = 6.8 Hz); LC-MS- 179.24

5-(4-Nitrophenyl)-1,2,4-triazolidine-3-thione (3b):
Yellow solid, Mp. 226-228 °C; 1H-NMR (500 MHz, DMSO-d6): δ 8.09 (s, 1H, CH), 8.11-8.13 (d, 2H, ArH, J = 10 Hz), 8.22-8.23 (d, 2H, ArH, J = 5Hz), 8.26 (s, 1H, NH), 8.41 (s, 1H, NH), 11.71 (s, 1H, NH); LC-MS- 223.1

5-(3-Nitrophenyl)-1,2,4-triazolidine-3-thione (3c):
Yellow solid, Mp. 226-228 °C; 1H-NMR (500 MHz, DMSO-d6): δ 7.67-7.70 (t, 1H, ArH), 8.15 (s, 1H, CH), 8.20-8.22 (dd, 1H, ArH, J = 10 Hz), 8.24-8.25 (d, 1H, ArH, J = 5 Hz), 8.31 (s, 1H, ArH), 8.33 (s, 1H, NH), 8.66 (s, 1H, NH)11.62 (s, 1H, NH); LCMS- 223.1

5-(4-Chlorophenyl)-1,2,4-triazolidine-3-thione (3d):
White crystal, Mp. 206-208 °C;1H-NMR (400 MHz, DMSO-d6): δ 7.46 (d, 2H, ArH, J = 8.4 Hz), 7.84 (d, 2H, ArH, J = 8.4 Hz), 8.02 (s, 1H, CH), 8.08 (s, 1H, NH), 8.24 (s, 1H, NH), 11.49 (s, 1H, NH) ; LCMS- 214.1

5-(4-Bromophenyl)-1,2,4-triazolidine-3-thione (3e):
White crystal, Mp. 202-204 °C; 1HNMR (400 MHz, DMSO-d6): δ 7.57-7.605 (d, 2H, ArH, J = 8.7 Hz),

7.75-7.78 (d, 2H, ArH, J = 8.4 Hz), 8.00 (s,1H, CH), 8.08 (s, 1H, NH), 8.24 (s, 1H, NH), 11.49 (s, 1H, NH); LCMS-258.1

5-(4-Fluorophenyl)-1,2,4-triazolidine-3-thione (3f):
White crystal, Mp. 172-174 ˚C; 1H-NMR (400 MHz, DMSO-d6): δ 7.23 (d, 2H, ArH, J = 8.4 Hz), 7.85 (d, 2H, ArH, J = 8.4 Hz), 7.88 (s, 1H, CH), 8.03 (s, 1H, NH), 8.20 (s, 1H, NH), 11.43 (s,1H, NH); LCMS-196.1

5-(o-Tolyl)-1,2,4-triazolidine-3-thione (3g):
White crystal, Mp. 174-176 ˚C; 1H-NMR (400 MHz,DMSO-d6): δ 2.37 (s, 3H, CH3), 7.18-7.22 (d, 2H, ArH, J = 8 Hz), 7.25-7.27 (d, 1H, ArH, J = 8 Hz), 7.89 (s,1H, ArH), 8.02-8.05 (t, 1H, ArH, J = 8 Hz), 8.202 (s, 1H, NH), 8.393 (s, 1H, NH), 11.34 (s, 1H, NH); LCMS-192.2

5-(4-Methoxyphenyl)-1,2,4-triazolidine-3-thione (3h):
White crystal, Mp. 156-158 ˚C; 1H-NMR (400 MHz, DMSO-d6): δ 3.78 (s, 3H, OCH3), 6.94-6.96 (d, 2H, ArH, J = 8.8 Hz), 7.71-7.74 (d, 2H, ArH, J = 7.6Hz), 7.92 (s, 1H, CH), 7.98 (s, 1H, NH), 8.11 (s, 1H, NH), 11.32 (s, 1H, NH); LCMS-210.1

5-(3,4-dichlorophenyl)-1,2,4-triazolidine-3-thione (3i):
White crystal, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.629-7.681 (d, 2H, ArH, J = 8 Hz), 7.703-7.729 (d, 1H, ArH, J = 8 Hz), 7.89 (s,1H, ArH), 8.02-8.05 (t, 1H, ArH, J = 8 Hz), 8.202 (s, 1H, NH), 8.393 (s, 1H, NH), 11.34 (s, 1H, NH); HRMS-247.918

5-(2-pyridinyl)-1,2,4-triazolidine-3-thione (3j):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.36-7.39 (d, 1H, ArH, J = 8 Hz), 7.80-7.84 (t, 1H, ArH, J = 8 Hz), 8.09 (s,1H, CH), 8.18 (s, 1H, NH), 8.27-8.29 (d, 1H, ArH, J=8Hz), 8.36 (s, 1H, NH), 8.55-8.57(d, 1H, ArH J =8Hz) 11.49 (s, 1H, NH); LCMS-179.1

5-(4-pyridinyl)-1,2,4-triazolidine-3-thione (3k):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (500 MHz, DMSO-d6): δ 7.78-7.79 (d, 2H, ArH, J = 5 Hz), 8.59-8.60 (d, 1H, ArH, J = 5 Hz), 8.01 (s,1H, CH), 8.22 (s, 1H, NH), 8.40 (s, 1H, NH), 11.69 (s,1H, NH); LCMS-179.1

5-(2-thienyl)-1, 2, 4-triazolidine-3-thione (3l):
Yellow solid, Mp. 174-176 ˚C; 1H-NMR (400 MHz, DMSO-d6): 7.098-7.119 (d, 1H, ArH, J = 12.6 Hz), 7.440-7.451 (d, 1H, ArH, J = 6.6 Hz), 7.557 (s,1H, CH), 7.644-7.656 (d, 1H, ArH J = 7.2Hz), 8.213 (s, 1H, NH), 8.234 (s, 1H, NH), 11.49 (s, 1H, NH); LCMS-184.1

III. RESULTS AND DISCUSSION

3.1 Catalytic activity of CuO NPs.

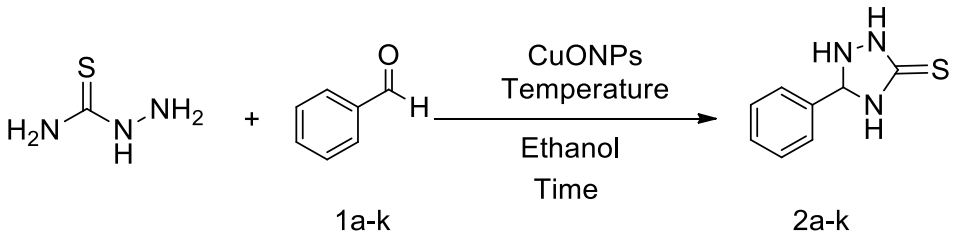
To optimize the reaction condition, the synthesis of 5-phenyl-1,2,4-triazolidine-3-thione (2a) from thiosemicarbazide and benzaldehyde was chosen as a model reaction in ethanol (Table 3.1).

Table 3.1 Effect of CuO NPs loading towards the synthesis of 5-phenyl-1,2,4 triazolidine-3-thione[(a)]

Entry	Amount of CuO NPs (in mg)	Time (in Min.)	Yield[(b)] (in %)
1	1	10	78
2	2	10	87
3	3	10	89
4	4	10	92
5	**5**	**10**	**95**
6	10	10	97

[a] Reaction conditions: 10 mmol Thiosemicarbazide, 10 mmol aromatic aldehyde, 5 mg of **CuO NPs**, 5 ml ethanol at 70 ^{0}C. [b] Isolated yield.

Table 3.2 CuO NPs catalyzed synthesis of 5-aryl-1,2,4-triazolidine-3-thione[(a)]



1a-k 2a-k

Entry	R	Time (Min.)	Product	Yield[(b)]
1	C_6H_5 2a	10	3a	95
2	4-(NO_2) C_6H_4 2b	6	3b	94
3	3-(NO_2) C_6H_4 2c	10	3c	91
4	4-(Cl) C_6H_4 2d	12	3d	94
5	4-(Br) C_6H_4 2e	14	3e	90
6	4-(F) C_6H_4 2f	20	3f	89
7	2-(Me) C_6H_4 2g	10	3g	94
8	4-(OCH_3) C_6H_4 2h	12	3h	95
9	3,4 (Cl) C_6H_3 2i	14	3i	90
10	2-Pyridine 2j	15	3j	93
11	4-Pyridine 2k	18	3k	95
12	2-Thiophene 2l	16	3l	94

[a] Reaction conditions: 10 mmol Thiosemicarbazide, 10 mmol aromatic aldehyde, 5mg of **CuO NPs**, 5 ml ethanol at 70 ^{0}C. [b] Isolated yield.

At first 1 mg of CuONPs were used that lead to 78 % of 2a at 70 ^{0}C within 10 mints (table 3.1 entry 1). On varying quantity of CuONPs (table 3.1 entries 2-5) yield of 2a were improved suggestively up to 95 %

presenting 5 mg of CuO NPs is enough for the transformation to 2a, further increment in amount of CuO NPs doesn't improve the yield of 2a (table 3.2 entry 6). After screening different solvents, the room temperature synthesis of 5-phenyl-1,2,4-triazolidine-3-thione improved yield were obtained with ethanol (Table 3.3)

Table 3.3 CuO NPs catalyzed synthesis of 5-aryl-1,2,4-triazolidine-3-thione[(a)]

Entry	Solvent	Amount of catalyst (mg)	Temperature (⁰C)	Time (Min.)	Yield
1	Isopropyl alcohol	5	70	10	88
2	Ethanol	5	70	10	97
3	Methanol	5	70	10	92
4	Water-ethanol	5	70	10	75

Thus, it may be claimed that aldehyde and thiosemicarbazide leading to imine intermediate at the same time undergoes cyclisation due to CuO NPs.

To check the practicability and generality of the CuO NPs, we have studied several substituted aldehydes (1a-l) proficient conversion into the corresponding 5-phenyl 1,2,4-triazolidine 3-thione (2a-l) in brilliant yield were obtained (table 3.2)

Electron donating or withdrawing group bearing aldehydes and five or six membered heterocyclic aldehydes containing one heteroatom were practically converted into the corresponding triazolidine-3-thiones with outstanding yields. Above results, state that there is no substituent effect[36]. The structures of synthesized compound were confirmed by DEPT, ^{1}HNMR, ^{13}CNMR and Mass analysis.

3.2 Reusability Studies

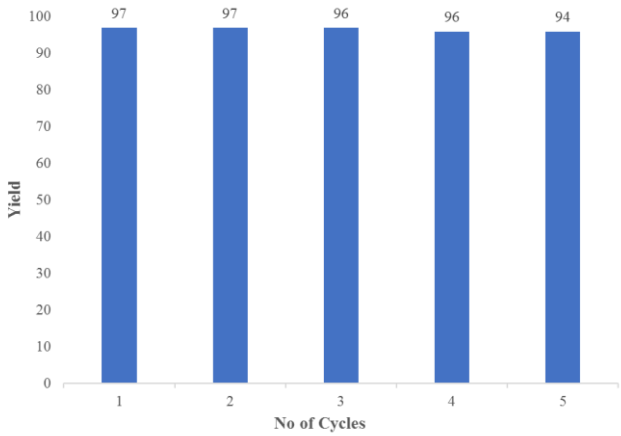


Figure 1. Reusability studies of CuO NPs for the synthesis of 5-phenyl-1,2,4-triazolidine-3-thione (2a)

The synthesis of 5-phenyl-1,2,4-triazolidine-3-thione from benzaldehyde and thiosemicarbazide using CuO NPs as catalyst under optimize reaction condition were adopted as model reaction for reusability study. As the reaction was completed ethyl acetate was added to the reaction mass and the catalyst was separated by centrifugation. Separated catalyst was washed with ethanol and acetone repeatedly, dried under vacuum and then reused for consecutive cycles. The efficiency of catalyst was studied up to five cycles without any significant loss. Figure. 5.8 presents reusability studies of CuO NPs.

IV. CONCLUSION

In present work, the synthesis of CuO NPs using aqueous extract of *Ziziphus mauritiana* L. leaves is developed. The catalytical study of CuO NPs towards the synthesis of 5-phenyl 1,2,4-triazolidine 3-thione derivative gives excellent yield, simple separation of catalyst by just centrifugation. The effectiveness of the catalyst was tested and the loss was negligible up to five cycles.

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