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Catalytic effect of nano copper ferrite on the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2-(1*H*)-one

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Abstract. The nano copper ferrite ($CuFe_2O_4$) catalyst was employed for the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2-(1*H*)-one by condensation and cyclization of 2-acetyl benzofuran, 9-anthracenaldehyde and urea under conventional heating reaction. The synthesized pyrimidinone was confirmed by physical constants, spectral (FT-IR, ^{1}H &^{13}C NMR) and elemental analysis. In this synthetic method, the authors investigated the effect of catalyst on the reaction by obtained yields.

***Keywords*:** copper ferrite; pyrimidine; eco-friendly synthesis; spectral studies.

1. Introduction

The first pyrimidine derivative alloxan (5,5-dihydroxypyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione) was discovered in 1818 by Brugnatelli by reacting uric acid with nitric acid [1]. Pyrimidines are aromatic heterocyclic six membered ring compounds containing two nitrogen atoms at the first and third positions [2]. Pyrimidine can be used as building blocks for the synthesis of π- conjugated materials [3, 4]. During the last two decades, aryl pyrimidine has been extremely studied as luminescent materials while ethyl pyrimidine remains less studied [5, 6]. These compounds are also widely used as application of biogenetic and photoactive materials [7, 8]. Pyrimidine derivatives can be synthesized by transition metal catalyzed methods [9, 10]. Pyrimidine derivatives are extremely useful as natural products, pharmaceuticals and functional materials [11, 12]. Several pharmaceutical essential compounds such as trimethoprim, sulfadiazine, Gleevec and vitamin B_1 are the different form of pyrimidines [13-15]. Pyrimidine moiety present in barbituric acid and its derivatives are employed as hypnotics [16]. These pyrimidine derivatives including 4,6-dimethyl pyrimidines are synthesized by the reaction between carbonyl compounds with urea or thiourea or guanidine or formamidine [17-19]. Pyrimidine has wide variety of biological actions such as antibacterial, antiallergic, antitumor, antifolate, tyrosine kinase, antimicrobial, anti-inflammatory, analgesic, antihypertensive, antileishmanial, tuberculostatic, anticonvulsant and anti-aggressive activities [20-24]. Thiamine and adenine are well known examples of readily available medications that contain feature pyrimidine moieties and are used to treat Parkinson disease [25]. Pyrimidines were widely used in coordination chemistry, as rich electron ligand that plays a role as interesting to form metal complexes [26-28].

Due to its utilization in sensors, environmental purification, magnetic and electric materials, ceramic coating, copper ferrite ($CuFe_2O_4$) has been widely developed [29]. Various synthetic methods adopted for preparing nano $CuFe_2O_4$ catalyst such as solid-state reaction, solvothermal method, chemical vaporization or decomposition, microwave-assisted method and sol-gel method [30]. The $CuFe_2O_4$ nanoparticles not only gives purity, high yield, and short time reaction but also it is a speedy, cheap, facile, and eco-friendly heterogeneous method to carried out the course of the reaction [31, 32]. Esmaeili et al. [33] reported the new synthesis of 62-89% yields of benzyl pyrazolyl pyrido[1,2-a] pyrimidine derivatives with nano copper ferrite catalyst under solvent free method. Souza et al. [34] and Anjaneyulu et al. [35] utilized the one-pot three component Biginelli reaction for the synthesis of dihydropyrimidines using benzaldehyde, urea or thiourea and acetoacetate or β-ketoester in presence of Brönsted acid catalysts under conventional heating method. Misra et al. [36] synthesized 63-78% yields of the pyrimidine derivatives of 1,5-benzodiazepines by one-pot synthesis through domino reaction under DABCO (1,4-diazabicyclo[2.2.2]octane) catalyst. Heravi et al. [37] adopted the solvent-free one pot synthetic method for the synthesis 90-99% yield of pyrimidine derivatives by the reaction of aldehyde, ketone and urea with sulfamic acid as a catalyst. Arulkumaran et al. reported the synthesis and spectral properties of some pyrimidine carboxamides by conventional heating of substituted dithioacetal and guanidine using acetic acid as a catalyst [38]. Senbagam et al. reported the conventional synthetic method, spectral characterization, QSAR

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study and antimicrobial actions of some pyrimidine Schiff's bases [39]. According to our knowledge, there is no report available in the literature for the catalytic impact of nano copper ferrite catalyzing agent on the synthesis of pyrimidin-2-one. Hence, the authors taken effort to investigate the catalytic activity of nano copper ferrite catalyst on the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl) pyrimidin-2(1*H*)-one.

2. Experimental

2.1. Materials and methods

The 2-acetyl benzofuran and essential chemicals were procured from Sigma Aldrich Chemical Company, Bengaluru-100, India. By using surface analytical techniques, the fundamental nano $CuFe_2O_4$ catalyst was identified. Agilent Cary 630N IR spectrophotometer (4000-400 cm^{-1}) was used for recording IR spectra using KBr as spectroscopic grade. For analyzing the Raman spectrum, Bruker RFS27 100 s^{-1} (1064 nm) spectrophotometer with 1024 × 256 pixels, liquid nitrogen-cooled germanium detector was employed. Doubly enhanced frequency neodymium doped yttrium aluminum garnet (Nd:YAG) was utilized for Raman investigation. The laser power was kept up to 15 mW for the sample. The FEI quanta FEG 250 high resolution scanning electron microscope was used for capturing HR-SEM pictures. Samples mounted gold platforms were taken for measuring pictures at various magnifications. The EDX measurement was executed on different spots of sample surface in order to reduce any potential variances due to diverse nature of the studied area. For detection of elements 0.1% concentration was used.

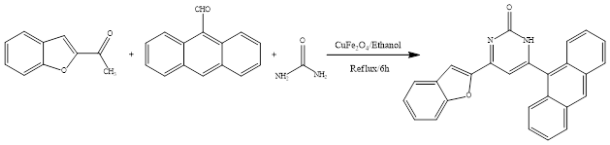
Melting point of synthesized pyrimidinone was taken in Raga Tech electrical melting point equipment and is uncorrected. IR spectrum of synthesized pyrimidine under evaluation was characterized using AGILENT CARY 630N IR spectrophotometer. The ^1H and ^{13}C NMR studies of synthesized pyrimidine under evaluation were detecting in BRUKER AVIII 400NMR spectrophotometer using 400 MHz for ^1H NMR and 125.46 MHz for ^{13}C NMR spectra. Deuterated chloroform was used as solvent combined with deuterated TMS as standard.

2.2. Preparation of $CuFe_2O_4$ catalyst

According to research article survey method was utilized for preparation of $CuFe_2O_4$ nano catalyst [29-31]. $CuFe_2O_4$ nano particles were prepared via co-precipitation method. About 75 mL of NaOH solution (4 M) was added to a solution of $Fe(NO_3)_3{\cdot}9H_2O$ (0.05 mol) and $Cu(NO_3)_2{\cdot}3H_2O$ (0.025 mol) at room temperature for 15 minutes. Then this was dissolved in 100 mL of distilled water to form a reddish-black precipitate. The reaction mixture was slightly heated at 90 ºC and stirred. It was cooled to normal temperature after 2-3 hours, and magnetic particles formed were separated using a magnetic separator. After that, the catalyst was washed with distilled water and kept in air oven over night at 80 ºC. Finally, the catalyst was ground in a mortar-pestle, kept in a furnace at 800 ºC and cooled to 100 ºC in air. The analyzed data of catalyst was well agreed with the data earlier reported in the literature. (See supplementary data for characterization of nano copper ferrite).

2.3. Nano copper ferrite assisted synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl) pyrimidin-2(1H)-one

One-pot three component conventional heating synthetic method was adopted for the synthesis of pyrimidine under the condensation cum cyclization of aldehyde, ketone and diamide. A mixture of 9-anthracenyl aldehyde (1 mmol), 2-acetyl benzofuran (1 mmol), urea (1 mmol), nano copper ferrite catalyst (0.35 mg) and 20 mL of ethanol were refluxed for 6 h (Scheme 1). TLC test was adopted for monitoring the completion of reaction condition. After running the reaction, the reaction product mixture was poured into ice water. The solid catalyst was separated by simple filtration. Evaporation of ethanol gave the reddish-brown precipitate of pyrimidinone. The crude product was further purified by recrystallization with ethanol. The recrystallized product was kept in a desiccator. The recovered catalyst was re-utilized after the catalyst was cleaned with ethyl acetate (8 mL) and hot air dried at 125 ºC in an oven for more than 1 h.

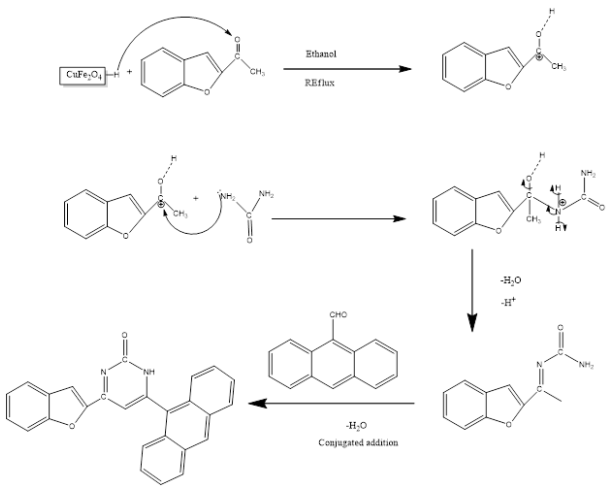


Scheme 1. The nano copper ferrite assisted synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl) pyrimidin-2(1*H*)-one by condensation cum cyclization.

3. Results and discussion

3.1. Catalytic effect of nano copper ferrite on the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1H)-one

In our organic chemistry research lab, we tried to synthesize the titled pyrimidinone by nano copper ferrite catalyst assisted condensation and cyclisation of 2-acetyl benzo furan, 9-anthracene aldehyde and urea under conventional heating method. Nano copper ferrite is the acidic in nature. This condensation undergoes acid catalyzed reaction mechanism [40]. The schematic diagram of the mechanism is shown in Scheme 2. The first step consists of protonation of carbonyl oxygen of 2-acetyl benzofuran by supplying of proton from acidic site of nano copper ferrite catalyst and carbonyl carbon gets positive charge. Second step consists of the nucleophilic attack of the positive carbon of carbonyl group of benzofuran by the amino group of urea then the nitrogen gets positive charge. Third step is the loss of removal of proton and water molecules leads to neutralize the positive charge of nitrogen atom and formed C=N. The fourth step is the conjugate addition of 9-anthracene aldehyde, cyclization and elimination of protons gave the pyrimidinone.



Scheme 2. The proposed mechanism of nano copper ferrite catalyzed conventional method for the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl) pyrimidin-2(1*H*)-one

The catalytic effect of nano copper ferrite catalyst was analyzed through the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one.

The quantity of catalyst was increased from 0.05 to 0.5 mg and the yield was improved from 30 to 87%. The ideal extent quantification of catalyst was considered as 0.35 mg. Beyond this quantity of catalyst there is no increase of yield in the reaction. This catalytic effect on the synthesis of pyrimidinone was illustrated in Figure 1.

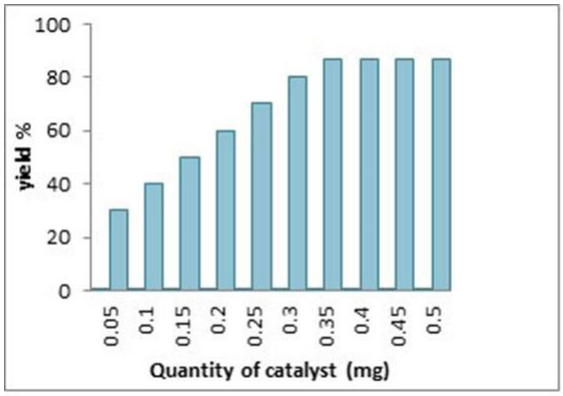


Figure 1. Catalytic effect on yield

Reusability of copper ferrite catalyst was investigated up to 6th run. The first 3 runs gave 87% yield. The 4, 5 and 6th runs gave 86% yields. Here there is no sustainable variation of yield in this reaction runs (Table 1).

Table 1. Reusability of nano copper ferrite catalyst for the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one

Run	1	2	3	4	5	6
Yield (%)	87	87	87	86	86	86

Also, the authors have studied the influence of the solvents on this reaction with the nano copper ferrite catalyst. The resultants of solvents on the yield of this reaction were investigated with different solvents such as ethanol, methanol, acetonitrile, dichloromethane, dioxane and tetrahydrofuran within the equal number of substrates, catalyst and 20 mL of solvents. In this experiment, the ethanol gave higher yield (87%) and dioxane gave the lowest yield (40%). The resultants of solvents on the yields were shown in Table 2.

Table 2. Effect of solvents on the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one with nano copper ferrite catalyst in conventional method

Solvent	ACN	DCM	EtOH	MeOH	THF	DO
Yield (%)	60	56	87	68	50	40

ACN: Acetonitrile; DCM: Dichloromethane; DO: Dioxane; EtOH: Ethanol; MeOH: Methanol; THF: Tetrahydrofuran

3.2. Spectral characterization of synthesized pyrimidinone

The synthesized 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one was identified by its physical constants, elemental analysis and spectroscopic data as: Reddish brown precipitate, yield: 87%, m.p. 112-115ºC. IR (KBr, ν_{max}, cm^{-1}): 3650 (pyrimidine N-H), 1654 (C=O), 3050 aromatic (C-H), 1577 (C=N), 1542 (C=C), 728 (NH_{op}). ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 5.67 ppm (S, 1H, C-H of pyrimidine ring), δ 8.08 ppm (S, 1H, N-H), δ 7.21-8.42 ppm (m, 9H, Ar-H). ^{13}C-NMR ($CDCl_3$, 400 MHz): δ 162.87 ppm (C=N), δ 156.46 ppm (C=O of pyrimidine ring), δ 102.89 ppm (hydrogen attached carbon), δ 111.2-134.12 ppm (Ar-C). M.F.: $C_{26}H_{16}N_2O_2$, M. Wt.: 388. Anal. (calcd.): C, 80.40 (80.39); H, 4.15 (4.16); N, 7.21 (7.23); O, 8.34 (8.36) (%). Mass analysis (m/z): 388.12[M^+] Calculated: 388.

The FT-IR spectra, ^{1}H-NMR and ^{13}C-NMR spectra of this compound are shown in Fig. 2-4.

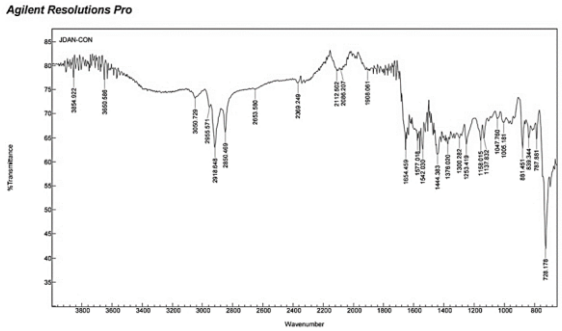


Figure 2. FT-IR spectrum of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one

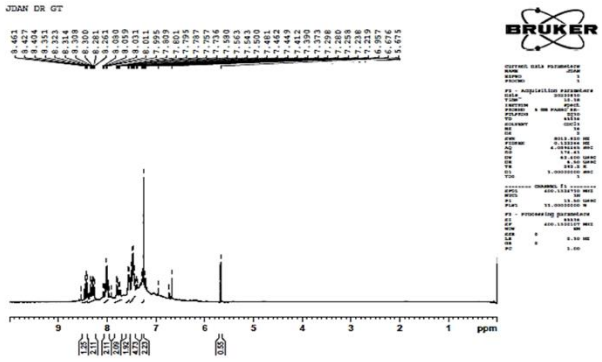


Figure 3. The ^{1}H-NMR spectrum of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one

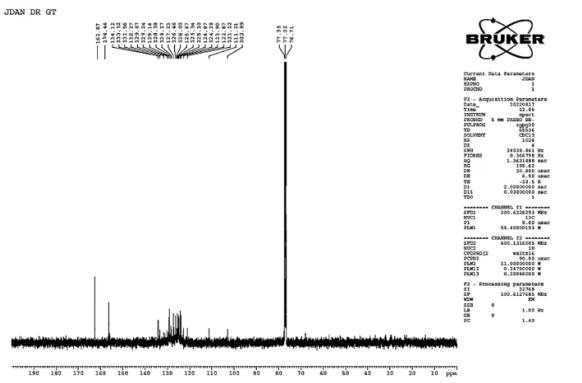


Figure 4. The ^{13}C-NMR Spectrum of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one

4. Conclusions

The catalytic effect of nano copper ferrite was identified by the synthesis of 6-(anthracen-9-yl)-4-(benzofuran-2-yl)pyrimidin-2(1*H*)-one under conventional heating method. From this experiment, the detected maximum yield was 87%. The reusability of the catalyst was proven to be good up to 6th runs of the reactions, considering that the observed yields did not change significantly. The ethanol solvent gave higher yield than other solvents. The synthesized pyrimidinone was identified using their physico-chemical parameters and spectroscopic data and are completely assisted for the synthesis of pyrimidine. Moreover, the nano copper ferrite catalyst was good and acceptable for the synthesis of pyrimidine via conventional heating method.

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Conflict of interest

The authors declare that there is no conflict of interest regarding this research article.

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