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Efficient synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes using zinc ferrite nanoparticles as a catalyst

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In this work we report an environmentally green and efficient zinc ferrite-nanocatalyzed protocol for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes by one-pot three-component condensation of aromatic aldehydes, malononitrile, and dimedone under reflux. The best yield was obtained by appropriate one-pot condensation reactions using different aromatic aldehydes with electron-donating or electron-withdrawing groups. Additionally, this catalyst is easily removed and can be recycled six times without considerable decrease in its activity. The advantages of this work include nanocatalyst reusability, short reaction time, non-toxic reaction, high purity, and excellent yield.

Keywords: Zinc ferrite nanoparticles, aromatic aldehyde, dimedone, malononitrile, 4*H*-chromenes, Knoevenagel condensation.

INTRODUCTION

Multicomponent reactions (MCRs) have an interesting function in organic and medicinal synthesis [1] consisting in combining three or more starting compounds to produce a final product. Almost all atoms in the starting compounds participate in the new final product [2-5]. In the last few years, MCRs have been powerful and effective bond forming mechanisms in organic and medicinal chemistry [6]. Typically, the MCRs technique gives an extensive amount of atom economy, convenience of a one-pot process, potential structural modifications, and a synthesis that takes less time and yields the target product [7]. The MCRs can save solvents and reagents, be used to synthesize complex compounds in a single conventional step and reduce complex purification processes due to the final product's inclusion of all starting materials [6, 7].

The synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene and its derivatives has recently generated a lot of interest due to their biological and pharmacological activities [8]. The 4*H*-chromene has many pharmacological properties, such as anticancer [9], diuretic [10], spasmolytic [11], anticoagulant [12], anti-anaphylactic [13], antitumor [14], cytotoxic [6], hypotensive [15], antibacterial [16], antiviral [17], antimalarial [18] and anti-anxiety [19]. 4*H*-chromenes are the fundamental structural component of a number of natural compounds. Furthermore, nitrile-functionalized 4*H*-chromene derivatives are helpful intermediates for the synthesis of a variety of chemicals, including lactones, pyridines, 1, 4-dihydropyridines, methyl acetoacetate, malonylurea, imido-esters, 1,3-cyclohexanedione, and aminopyrimidines [20-22]. In addition, other applications can be mentioned, such as lasers, dyes, pigments, cosmetics, optical brighteners, and biodegradable agrochemical products [20, 21].

In this work, the synthesis of 4*H*-chromene derivatives was performed using zinc ferrite nanoparticles. The mixture of aldehyde, malononitrile, and dimedone was used in the presence of various catalytic systems such as poly(4-vinylpyridine) [1], WETSA [11], Zn(L-proline)$_2$ [7], HDMBAB [12], Yb(PFO)$_3$ [8], β-CD-glycerin [16], SBSSA [6], MCM-41@Schiff base-Co(OAc)$_2$ [23], Fe_3O_4@SiO_2@propyl-ANDSA [17], β-CD [13], Fe_3O_4@GO-N-(pyridin-4-amine) [15], RGO-Pr-SH@AuNPs [24], $BaFe_{12}O_{19}$@IM [21], HMS/Pr-Rh-Zr [25], Bis-Su [26], aspartic acid [27], CuO nanoparticles [28], Fe_3O_4@UiO@DAS [29], and CaO@SiO_2-SO_3H [30].

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However, most of these reactions suffer from several drawbacks like expensive catalyst, toxic metal, drastic reaction conditions, long reaction time and low yields. Hence, there is still a demand for ecofriendly, efficient, feasible, and cost-effective synthetic protocols for the synthesis of 4*H*-chromenes. In this respect, significant efforts are being made to give out the hazardous catalysts for cheap, safe, and environmentally friendly biodegradable alternatives [20]. As we continue our long-term research focused on creating more advanced synthetic methodologies, herein we would like to share a simple green method for the efficient synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives.

EXPERIMENTAL

Material

The 4*H*-chromene products were identified by comparing their spectral, analytical, and physical properties to those published in the literature. The physical constants were determined in an open capillary. The progress of the reaction was monitored by thin-layer chromatography on silica gel-coated ALUGRAM SIL G UV254 plates. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance NEO 500 MHz spectrometer using DMSO-d_6 as a solvent. A mass spectrum was recorded on a SYNAPT DB A064 spectrometer.

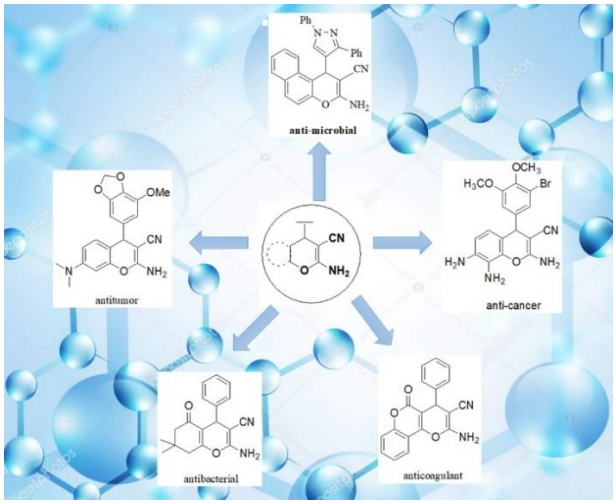


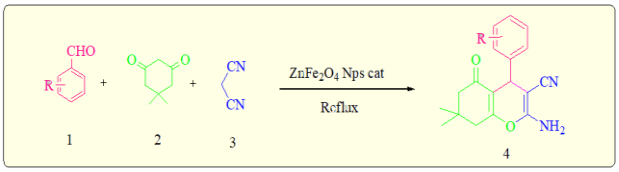
Figure 1. Some bioactive molecules containing chromene derivatives

Synthesis of zinc ferrite nanocatalyst

The zinc ferrite nanoparticles were synthesized by the reported method [31]. The zinc ferrite nanoparticles were characterized by FT-IR, XRD, SEM, TEM, EDAX, TG-DTA, and BET techniques [31].

General procedure for synthesis of 5-oxo-5,6,7,8-tetrahydro-4H-chromene derivatives

A mixture of aromatic aldehyde (1 mmol), malononitrile (1 mmol), dimedone (1 mmol) and zinc ferrite nanoparticles (0.04 g) as a catalyst was used. Then ethanol (10 ml) was added to the above reaction mixture, and the mixture was refluxed. The progress of the reaction was followed by thin-layer chromatography using n-hexane:ethyl acetate (4:1) as an eluent. After completion of the reaction, the crude product was filtered and dried, then recrystallized from ethanol to afford a pure product. The excellent purity of the product was confirmed by comparing physical and spectroscopic data (^{1}H NMR, ^{13}C NMR, and MS) to actual samples reported in the literature.



Scheme 1. Synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives

Spectral data of some 4H-chromene derivatives

- *2-amino-7, 7-dimethyl-5-oxo-4-phenyl-5, 6, 7, 8-tetrahydro-4H-chromene-3-carbonitrile (4a)*: Colorless crystal; m.p. 230-232°C; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 0.95-0.99 (s, 3H, CH_3), 1.03-1.07 (s, 3H, CH_3), 2.08-2.26 (d, 1H, CH, *J = 16.3 Hz*), 2.23- 2.26 (d, 1H, CH, *J = 16.3 Hz*), 2.49-2.51 (s, 2H, CH_2), 4.17 (s, 1H, CH), 6.98-7.16 (s, 2H, NH_2), 7.17- 7.19 (m, 3H, ArH, *J = 5.7 Hz*), 7.26-7.29 (d, 2H, ArH, *J = 5.4 Hz*); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm); 26.70, 28.29, 31.69, 35.48, 49.88, 58.24, 112.65, 119.60, 126.46, 127.04, 128.22, 144.63, 158.40, 162.38, 195.53; MF = $C_{18}H_{18}N_2O_2$, MS; Calc. [M] m/z = 294.35; Obs. m/z = 295.16 $(M+H)^+$.
- *2-amino-4-(4-methoxyphenyl)-7, 7-dimethyl -5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4b):* White crystal; m.p. 194-196°C; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 0.94 (s, 3H, CH_3), 1.03 (s, 3H, CH_3), 2.07-2.10 (d, 1H, CH, *J = 16.08 Hz*), 2.22-2.25 (d, 1H, CH, *J = 16.08 Hz*) 2.48-2.50 (m, 2H, CH_2), 3.71 (s, 3H, OCH_3), 4.11 (s, 1H, CH), 6.82-6.84 (d, 2H, ArH, *J = 8.64 Hz*), 6.93 (s, 2H, NH_2), 7.03 (d, 2H, ArH, *J = 8.60 Hz*); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm); 27.69, 28.88, 32.20, 34.76, 40.69, 50.70, 55.22, 63.82, 113.99, 114.25, 118.74, 128.63, 135.47, 157.38, 158.62, 161.26, 195.99; MF = $C_{19}H_{20}N_2O_3$, MS; Calc. [M] m/z =347; Obs. m/z =347 $(M+H)^+$.
- *2-amino-4-(4-bromophenyl)-7, 7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4c):* White crystal; m.p. 212-214°C; ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 0.94 (s, 3H, CH_3), 1.03 (s, 3H, CH_3), 2.08-2.12 (d, 1H, CH, *J= 16.08 Hz*), 2.23-2.26 (d, 1H, CH, *J= 16.04 Hz*) 2.50-

2.51 (m, 2H, CH_2), 4.18 (s, 1H, CH), 7.04 (s, 2H, NH_2), 7.10-7.12 (d, 2H, ArH, *J = 8.36 Hz*), 7.47-7.48 (d, 2H, ArH, *J = 8.24 Hz*); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm); 26.93, 28.37, 31.88, 35.27, 50.02, 57.86, 112.31, 119.66, 119.72, 129.59, 131.31, 144.22, 158.58, 162.86, 196; MF = $C_{18}H_{17}N_2O_2Br$, MS; Calc. [M] m/z =373.24; Obs. m/z =373 $(M+H)^+$.

Application of $ZnFe_2O_4$ nanocatalyst for synthesis of 5-oxo-5,6,7,8-tetrahydro-4H-chromene derivatives

In the present work, the $ZnFe_2O_4$ nanocatalyst [28] was successfully utilized for the one-pot synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives using substituted aromatic aldehydes, malononitrile, and dimedone under reflux conditions (Scheme 1). A model reaction of aromatic aldehydes (1 mmol), malononitrile (1 mmol) and dimedone (1 mmol) was used for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives (4a). Initially, the reaction conditions were optimized, including solvent, temperature, and time, as shown in Table 1. To identify the most effective solvent for this transformation, the model reaction was performed using different solvents like H_2O, EtOH and EtOH:H_2O (1:1) under different conditions. The reaction performed as indicated above with ethanol under reflux conditions was considered most suitable for the successful synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes.

Table 1. Optimization of reaction conditions for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes

Entry	Condition	Temp. (°C)	Time (min)	Yield (%)
1	H_2O	r.t	180	No reaction
2	H_2O	60-80	120	No reaction
3	H_2O	reflux	75	24
4	EtOH:H_2O	60-70	90	28
5	EtOH:H_2O	reflux	75	49
6	EtOH	r.t	60	34
7	EtOH	60-80	60	47
8	EtOH	reflux	10	74
9	EtOH	reflux	15	88
10	EtOH	reflux	20	92
11	EtOH	reflux	30	95
12	EtOH	reflux	40	95

Reaction conditions: aromatic aldehyde (1 mmol), dimedone (1 mmol) and malononitrile (1 mmol) in presence of $ZnFe_2O_4$ nanoparticles.

The amount of catalyst has played an important role in the reaction. In the absence of a catalyst, the reaction did not proceed. The results showed that as the amount of catalyst increases, the amount of product also increases up to 0.04 g of $ZnFe_2O_4$ nanoparticles (Table 2). All of these reactions were completed in a reaction time of around 30-60 min with good yields of product 4(a-n). In a similar way, the most suitable condition for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives, was found to be using 0.04 g of catalyst under reflux conditions.

Table 2. Optimization of catalyst in the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes

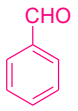
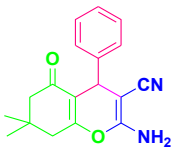
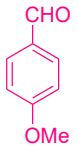
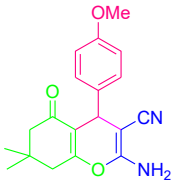
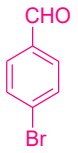
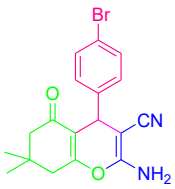
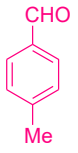
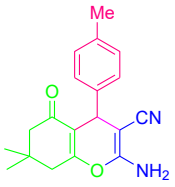
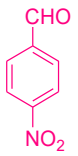
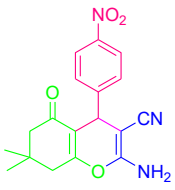
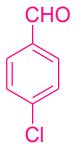
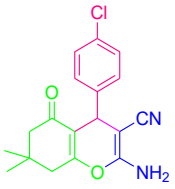
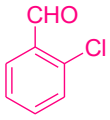
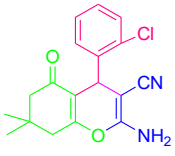
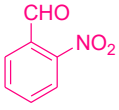
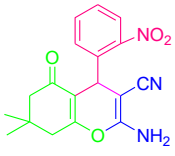
Entry	Amount of catalyst (g)	Reaction time (min)	Yield (%)
1	0	40	0
2	0.010	20	56
3	0.020	20	68
4	0.030	20	79
5	0.040	20	92
6	0.040	30	95
7	0.050	40	94

Reaction conditions: aromatic aldehyde (1 mmol), dimedone (2 mmol) and malononitrile (1 mmol) in presence of $ZnFe_2O_4$ nanoparticles in ethanol reflux.

We analysed the broadness, efficiency, variety, and scope of the reactions between aromatic aldehydes, malononitrile, and dimedone with zinc ferrite nanoparticles as a catalyst under reflux conditions in the presence of ethanol. The catalytic function of zinc ferrite nanoparticles was investigated using multiple aromatic aldehydes having -Me, -OMe, -NO_2, -Cl, -OH, -Br, -F groups (see Table 3). The present results were expanded to include several aromatic aldehydes with substituents donating and withdrawing electrons, including methyl, nitro, hydroxyl, methoxy, halogens and so on (Table 3). All reactions were performed to obtain the corresponding 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes with good product yield (86-96 %). The findings in Table 3 show that the yields of the products were not affected by the electronic effect of the various substituents on the aromatic aldehydes. With both (electron-donating/withdrawing) substituents, all of the reactions were similarly active at the positions of ortho, meta, and para on the aromatic aldehyde (Table 3).

As a way to show its suitability, the selected procedure was compared with the methods reported in Table 4. The safe and cost-effective zinc ferrite nanoparticles have high catalytic activity, excellent result in terms of reaction conditions, short reaction time, and good yield, suitable for a wide range of aldehydes.

Table 3. Synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes using $ZnFe_2O_4$ nanoparticles

Entry	Substrate	Product	Time (min)	Yield (%)	M.P. (°C)		Ref.	TON	TOF
					Obs.	Rep.			
4a			30	95	230-232	229-231	[32]	5.7	11.4
4b			30	96	194-196	193-195	[9]	5.8	11.6
4c			55	92	212-214	210	[16]	5.5	6.04
4d			35	94	210-212	210	[33]	5.6	9.6
4e			35	86	172-174	174-176	[34]	5.2	8.9
4f			30	93	210-212	208-210	[35]	5.6	11.2
4g			40	92	202-204	201-203	[36]	5.5	8.3
4h			45	93	230-232	228-230	[10]	5.6	7.4

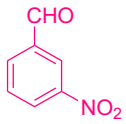
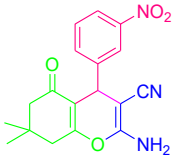
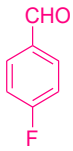
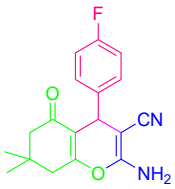
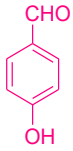
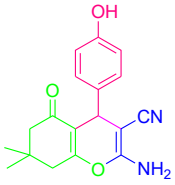
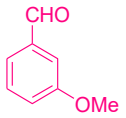
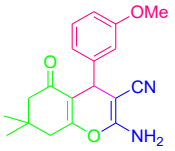
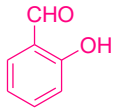
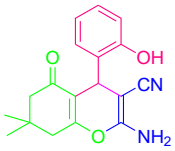
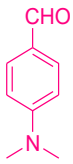
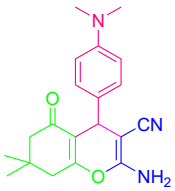
4i			35	89	208-210	210-211	[37]	5.3	9.1
4j			35	88	188-190	186-189	[1]	5.3	9.1
4k			40	90	216-218	215-220	[16]	5.4	8.1
4l			35	94	188-190	187-189	[9]	5.6	9.6
4m			40	90	184-186	185-186	[36]	5.4	8.1
4n			60	92	200-202	202-204	[8]	5.5	5.5

Table 4. Comparison of catalytic efficiencies for synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes

Entry	Catalyst	Solvent	Temp. (°C)	Time (min)	Yield (%)	Reference
1	$Zn(L\text{-proline})_2$	$EtOH:H_2O$ (2:3)	reflux	3h	85	[7]
2	HDMBAB	H_2O	80-90	5h	92	[12]
3	$Yb(PFO)_3$	EtOH	60	5h	90	[8]
4	β-CD (aq. glycerine)	H_2O	35-40	2h	90	[16]
5	SBSSA	$EtOH:H_2O$ (1:1)	reflux	75	94	[6]
6	$L_4/Co(OAc)_2$	H_2O	50	3h	94	[23]
7	B-CD	H_2O	r.t	5h	92	[13]
8	Aspartic acid	$EtOH:H_2O$ (1:1)	r.t	1h	93	[27]
9	$ZnFe_2O_4$ nanoparticles	EtOH	reflux	30	95	Present work

Proposed mechanism

A plausible mechanism for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes using zinc ferrite nanoparticles is shown in Figure 2. In the first step is the Knoevenagel condensation by the reaction of aromatic aldehyde with malononitrile. Also, we believe that the cyanide group of intermediates is activated by the zinc ferrite nanocatalyst for the nucleophilic attack of dimedone to form a Michael adduct. The intermediate undergoes ring closure and tautomerization yielding the desired products.

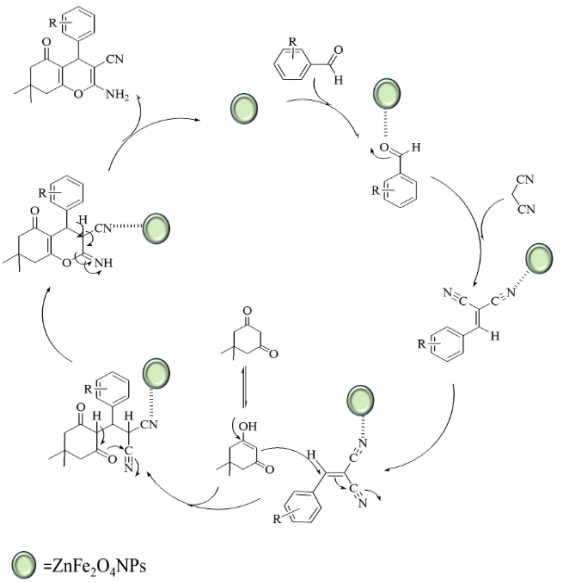


Figure 2. Plausible reaction mechanism for synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromenes

CONCLUSION

In conclusion, in this study we have developed a very simple, efficient and green methodology for the synthesis of 5-oxo-5,6,7,8-tetrahydro-4*H*-chromene derivatives in the presence of $ZnFe_2O_4$ nanoparticles. The availability of the catalyst, easy reaction, simple setup, short reaction time, and excellent product yields are the most important features of this study.

The FT-IR, XRD, SEM, TEM, EDAX, TG-DTA, BET, NMR and MS data are in the Supplementary file**.**

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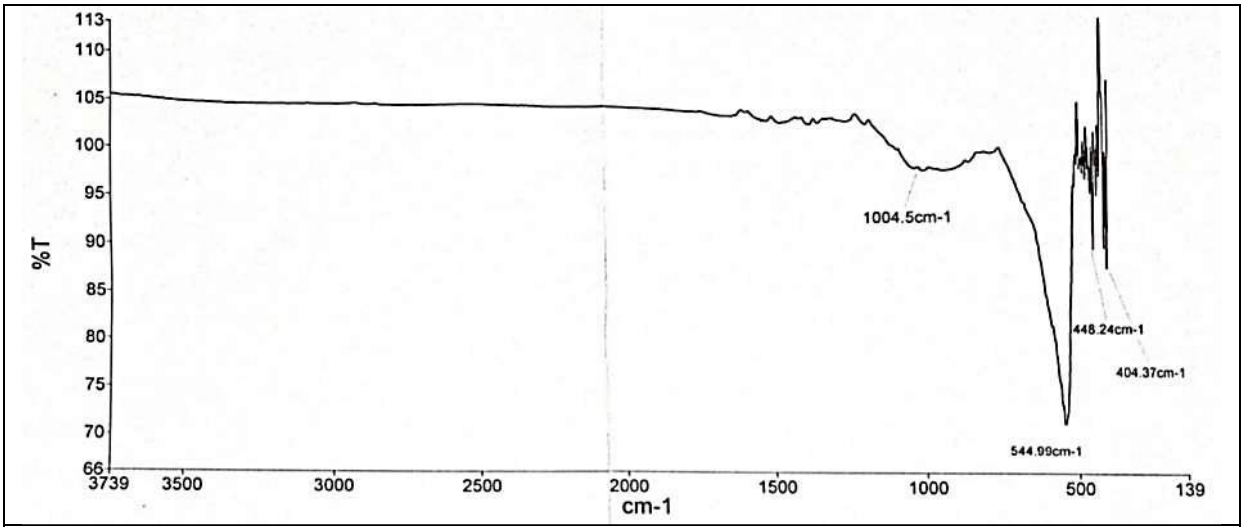
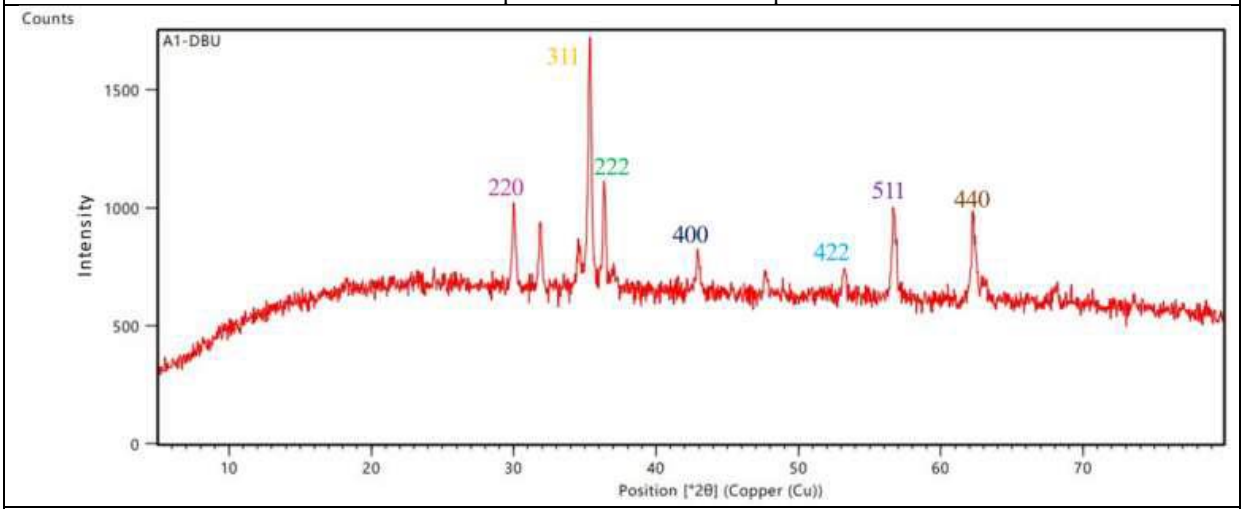
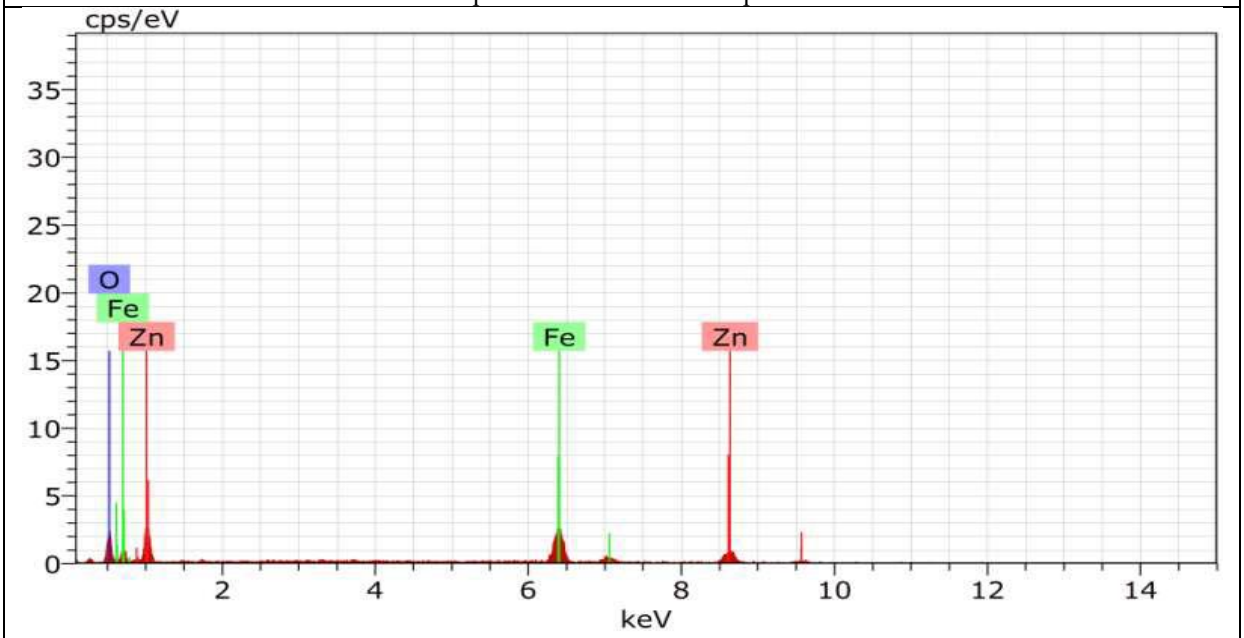


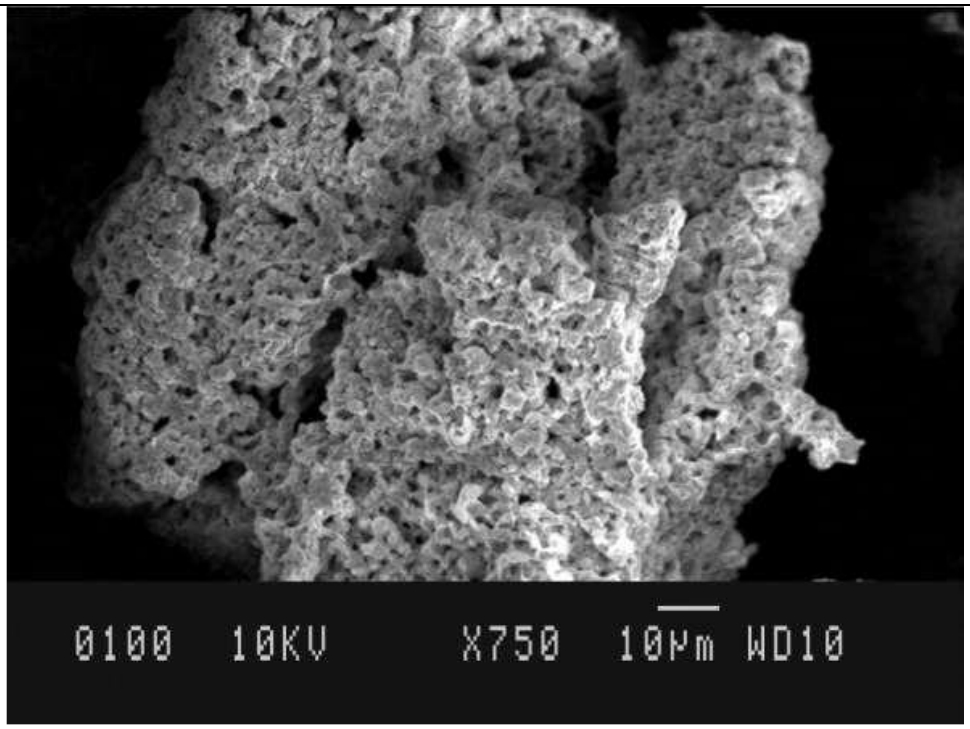
FTIR spectra for ZnFe2O4 nanoparticles



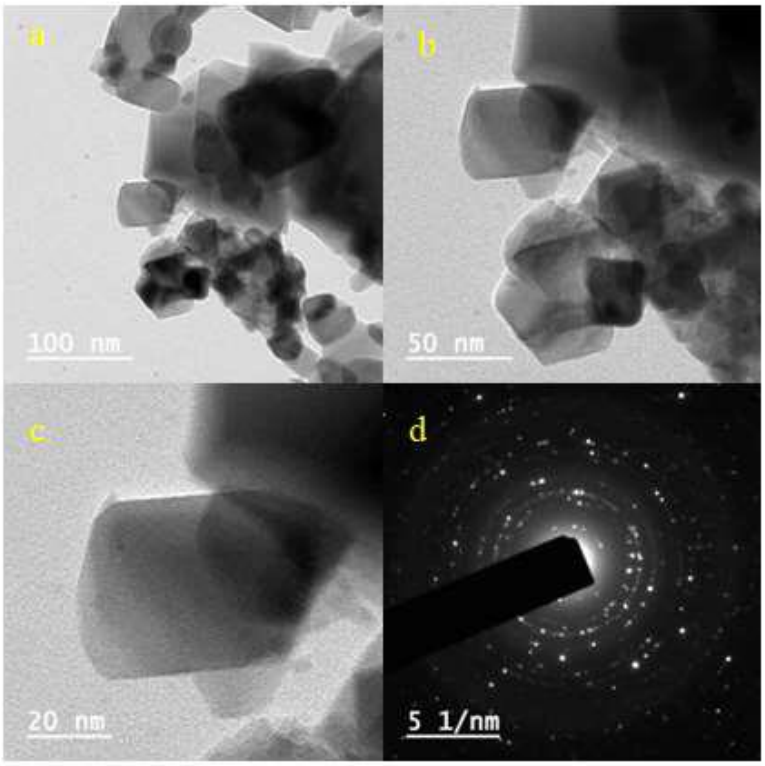
XRD pattern of ZnFe2O4 nanoparticles



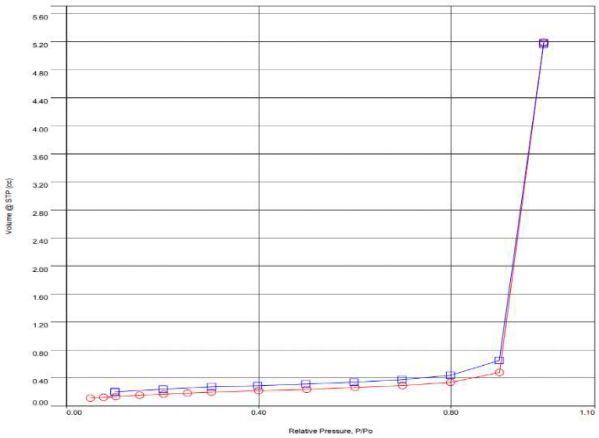
EDAX spectrum of ZnFe2O4 nanoparticles



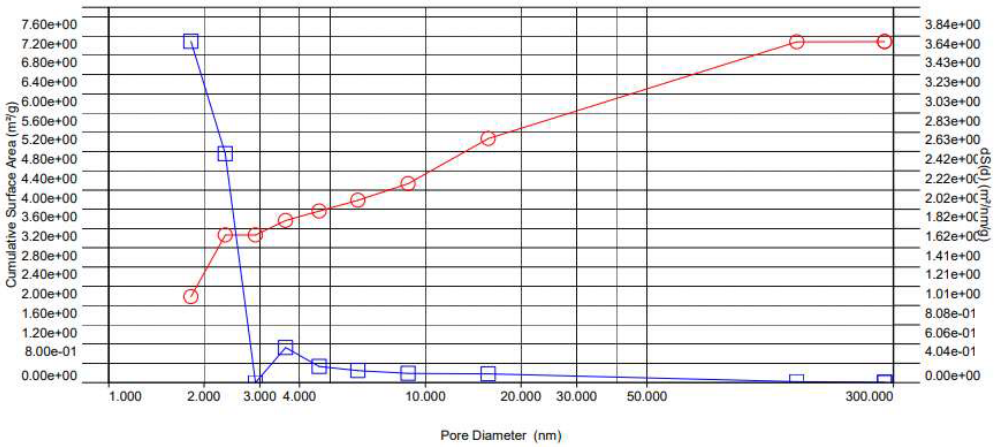
SEM image of ZnFe2O4 nanoparticles



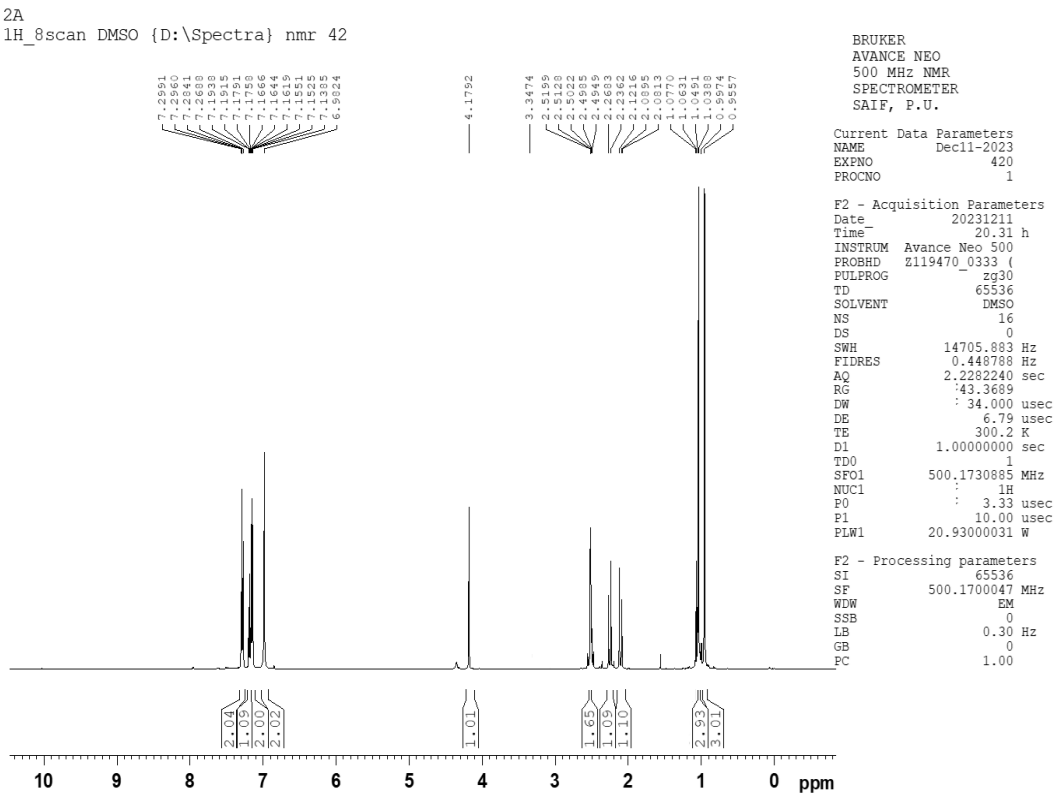
FETEM of ZnFe2O4 nanoparticles



BET spectrum of ZnFe2O4 nanoparticles: BET N2adsorption-desorption curves



BET spectrum of ZnFe2O4 nanoparticles: Average pore radius for ZnFe2O4nanoparticles



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