



Magnetically Recyclable Silver nanoparticles coated with Nano Fe₂O₃ Catalyst for Multicomponent Synthesis of 1,8-dioxodehydroacridine Derivatives under solvent-free conditions

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Abstract: A new and efficient technique for creating 1,8-dioxodehydroacridine derivatives has been established in this work. This method used cyclic 1,3-diketone, aldehydes, and an ammonium acetate in a one-pot condensation process. The reaction was environmentally benign because it was conducted without the use of solvents and at room temperature. Ag-Fe₂O₃, a catalyst with a core-shell nanostructure, was used in this procedure. The synthesis of the intended derivatives was made easier and more effective by the application of this catalyst. Large-scale product production, quick reaction kinetics, straightforward purification processes, and easy implementation are only a few benefits of the current technology.

Keywords: Ag coated with Fe₂O₃; core-shell nanostructures; magnetically separable catalyst; Acridine derivatives.

I. INTRODUCTION

Acridines are a type of nitrogen-containing fused heterocyclic compounds with a distinctive structural makeup and a wide variety of biological functions. Numerous investigations into their synthesis, reactivity, and pharmacological potential have been prompted by their distinct molecular structure, which is defined by a tricyclic ring system.[1–3].

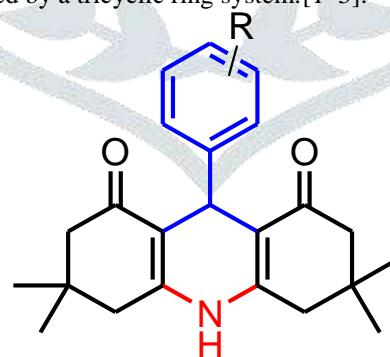


Fig 1: General Structure of Acridine

Acridine and its derivatives are widely used in various industrial applications [4]. These compounds have been known since the 19th century and were initially utilized as pigments and dyes [5]. Thus far, a variety of chemical and physical properties have been observed in acridine derivatives [6]. Their efficacy in the pharmaceutical sector has also been documented. The synthesis of acridine derivatives is a crucial and fundamental objective in the field of organic chemistry [7]. There has been a notable emphasis on the fabrication of 1,8-dioxodehydroacridines owing to their noteworthy physiological and biological functionalities, encompassing activities such as: Antimicrobial [8], Anti-tumor [9], Anti-inflammatory [10], Cytotoxic [11], Analgesic [12], Anti-fungal [13]. The compounds currently under investigation are frequently synthesized via a tricomponent reaction, wherein 1,3-diketone, aldehydes, and a range of anilines or ammonium acetate are employed. The reaction under consideration is commonly catalysed by a variety of catalysts, including Carbon-Based Solid Acid [14], PPA-SiO₂ [15], Ammonium Chloride [16], Polyvinylpolypyrrolidone-supported boron trifluoride [17], PPA/Al₂O₃ [18], Amberlite IR-120H [19], Cu(II) Schiff Base [20], Cu-Doped ZnO [21] and proline [22]. However, it's crucial to remember that a number of approaches that have been previously documented in the literature have built-in

limitations. These restrictions include lengthy reaction times, stoichiometric reagent requirements, costly catalysts, low product yields, the use of hazardous solvents, the application of traditional work-up procedures, challenges with catalyst separation, and the incapacity to recover the catalyst. [23–29]. The identification of innovative techniques employing new heterogeneous and reusable catalysts remains essential to improve the reaction conditions for synthesizing 1,8-dioxodehydroacridines and to address the related constraints. [30, 31]. In this section we have discussed the facile and efficient one pot condensation of aldehydes, dimedone, and either ammonium acetate or aniline to produce 1,8-dioxodehydroacridine derivatives in high yields (Fig 2). This is achieved through the utilization of a nanoscale, environmentally-friendly magnetic core-shell nanocatalyst composed of $\text{Ag@Fe}_2\text{O}_3$, which was synthesized by simple sol gel auto-combustion method. This approach is cost-effective and environment friendly as it involves a one-step synthesis that is facile to handle and does not necessitate harsh conditions like high temperature and strong reactants.

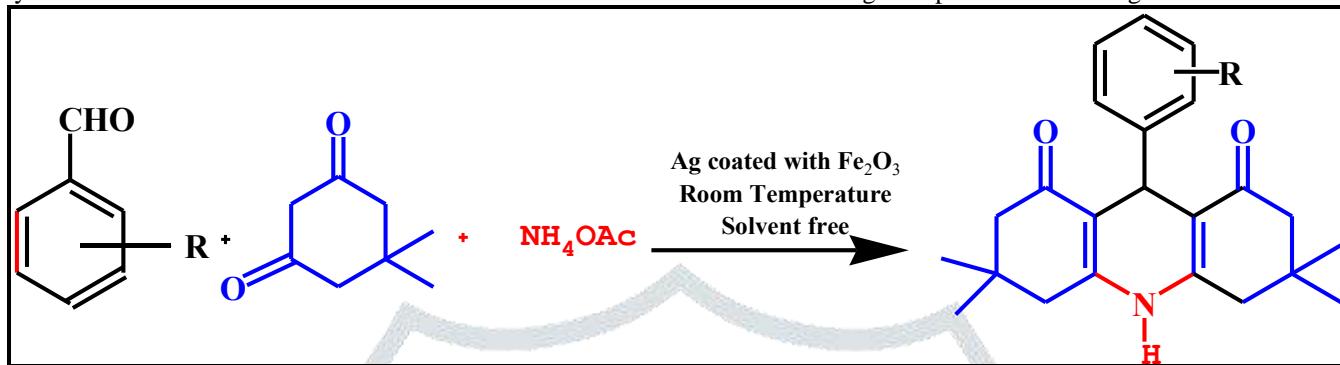


Fig 2: Ag coated with Fe_2O_3 catalysed synthesis of 1,8-dioxodehydroacridine derivatives

II. MATERIALS AND METHODS

The chemicals and reagents used in the study are of AR grade; they were purchased from SD Fine Chemicals and were utilized without further purification. Deionized water was used to prepare all of the solutions.

2.1 Synthesis of Ag coated with Fe_2O_3 Core-Shell Nanoparticles

Ag coated with Fe_2O_3 core-shell nanoparticles were synthesized by sol-gel auto-combustion method [37]. Ferric Nitrate Nonahydrate (2mmol), Silver Nitrate (1mmol), and Citric Acid (1.5 mmol) were used as precursors. In the typical preparation method, all precursors were mixed with vigorous stirring. After 10 minutes of stirring, ammonia solution was added dropwise to adjust the pH of the solution to 12. The stirring was continued for another 1h at an elevated temperature (60 °C). The temperature was then slowly raised to 100°C until the solution turns into a viscous gel, which, on further heating, undergoes auto combustion to form a black-coloured powder of Ag coated with Fe_2O_3 nanoparticles. Various techniques were used to characterize the as-prepared material.

2.2 Characterization of Ag coated with Fe_2O_3 Catalyst

The formation of Ag coated with Fe_2O_3 nanoparticles was confirmed by X-ray diffraction patterns recorded using Bruker D8 Advance using Cu $\text{K}\alpha$ radiation (wavelength $\lambda = 0.15406$ nm) over diffraction angles $2\theta = 20\text{--}80^\circ$. Fourier transform infrared spectroscopic (FT-IR) (Spectrum one: FT-IR Spectrometer) analysis was performed between 450–4000 cm^{-1} to investigate the information regarding the chemical bonds. The surface morphology of the nanoparticles was studied using Field Emission Scanning Electron Microscopy (FE-SEM), EDAX mapping (JEOL 6390 LA/ OXFORD XMX N). The magnetic studies were conducted using Vibrating Sample Magnetometer (Lakeshore VSM 7410). Thermal behaviour was studied using TG/DSC techniques (NETZSCH STA 449 F3 Jupiter). The Zeta potential of Ag coated with Fe_2O_3 was determined using Malvern Zeta Sizer. The specific surface area was calculated by using the single-point dynamic N_2 BET method at -196°C with N_2 purging for one hour with Micromeritics ASAP 2010.

2.3 Synthesis of 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione:

A mixture of aromatic aldehyde (1 mmol), dimedone (2 mmol), and ammonium acetate (1 mmol) is heated in the presence of a catalytic amount of Ag coated with Fe_2O_3 (10 mg) under solvent-free conditions at room temperature for the stipulated time. The progress of the reaction was monitored by TLC (*n*-Hexane: EtoAc, 9:1). The final product was heated in ethanol and poured into water. The catalyst was magnetically removed and washed with chloroform for reuse. The residue was then poured into crushed ice and stirred. The pure product was obtained by recrystallization from hot aqueous alcohol. This resulted in the production of 3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione with a yield of 89–98% (Table 1). The purified products were confirmed from the melting points of the synthesized compounds, were determined by the open capillary method, and were reported without any further correction. Fourier Transform Infrared (FT-IR) spectrum of the products was recorded using a 3000 Hyperion Microscope with Vertex 80 FTIR System (Bruker, Germany) to determine the presence of specific functional groups in the molecule. ^1H and ^{13}C NMR spectra were performed in CDCl_3 using TMS as an internal standard on a 400 MHz liquid state NMR spectrometer instrument. All Proton chemical shifts (δ) are relative to tetramethylsilane (TMS, $\delta = 0.00$) as an internal standard and coupling constants (J) are given in Hz.

2.4 Spectral data of representative organic molecules:

Table 1, Entry 2: IR (KBr) ν : 3215, 2957, 1730, 1590, 1486, 1339, 1219, 832, 751 cm^{-1} ; ^1H NMR (400 MHz, DMSO) δ = 8.44 (s, 1H), 7.95 (d, J =7.4, 2H), 7.50 (d, J =7.4, 2H), 4.45 (s, 1H), 2.43 (s, 4H), 1.68 (s, 4H), 1.09 (s, 12H); ^{13}C NMR (100 MHz, DMSO) δ = 199.48, 194.51, 165.01, 159.55, 149.32, 145.68, 134.28, 128.99, 123.19, 112.54, 65.31, 51.49, 36.36, 32.86, 32.71, 28.81.

Table 1, Entry 3: IR (KBr) ν : 3166, 2955, 2870, 1714, 1577, 1507, 1467, 1368, 1219, 1036, 833, 749 cm^{-1} ; ^1H NMR (400 MHz, DMSO) δ 8.27 (s, 1H), 7.16 (d, J = 7.4 Hz, 2H), 6.71 (d, J = 7.6 Hz, 2H), 4.42 (s, 1H), 2.05 (s, 4H), 1.61 (s, 4H), 1.11 (s, 12H); ^{13}C NMR (100 MHz, DMSO) δ = 194.68, 185.05, 159.64, 157.19, 129.32, 129.21, 115.15, 113.54, 91.34, 53.49, 36.40, 32.99, 32.62, 27.90.

III. RESULT AND DISCUSSION

The present work is based on the investigation of the catalytic efficiency of Ag coated with Fe_2O_3 core-shell nanoparticles which were prepared using the sol-gel auto-combustion technique in a single step.

3.1 Characterization of catalyst

Fig 3 represents the XRD pattern of Ag coated with Fe_2O_3 . It exhibits the characteristic peaks of the crystalline phase of Ag together with $\gamma\text{-Fe}_2\text{O}_3$. According to the JCPDS reference powder diffraction files, the diffraction peaks indexed to the cubic spinel phase of Ag (JCPDS 65-2871) and $\gamma\text{-Fe}_2\text{O}_3$ (JCPDS 39-1346). The absence of additional peaks indicates the formation of high purity nanoparticles. The strong intensity peaks at 38.12° , 44.32° , 64.44° and 77.41° correspond to (111), (200), (220), and (311) diffraction planes of Ag core. The weak peaks for $\gamma\text{-Fe}_2\text{O}_3$ in the diffraction pattern suggest the formation of a thin shell of Fe_2O_3 over Ag-core, which agrees EDS mapping as well. The average crystallite size was calculated using the Scherrer formula, which was found to be 73.7 nm.

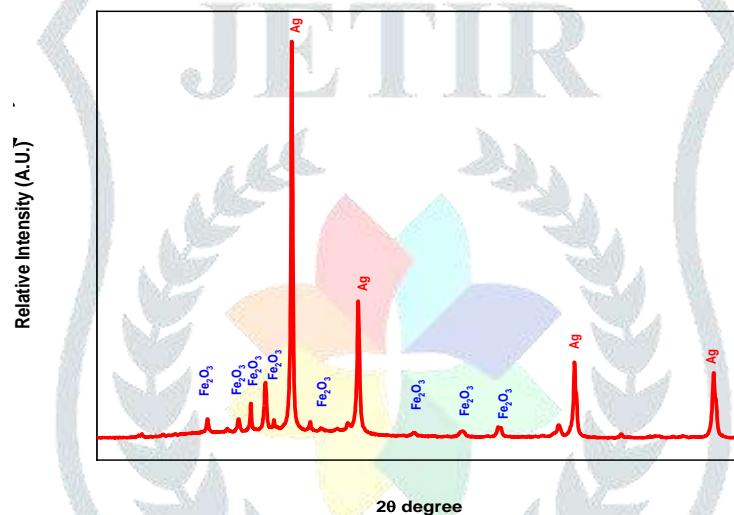


Fig. 3: XRD patterns of Ag coated with Fe_2O_3

Figs. 4a and 4b are the FTIR spectra of Fe_2O_3 and Ag coated with Fe_2O_3 nanoparticles, respectively. The FTIR spectra of bare Fe_2O_3 showed stretching frequencies of 441, 553, and 628 cm^{-1} , respectively, which were attributed to Fe–O bond ($\text{Fe}^{2+3+}–\text{O}^{2-0}$ occupying octahedral and tetrahedral sites) [38]. A slight shift in the location of bands at 454 cm^{-1} and 533 cm^{-1} in fig. 4b attributes to an imaginable contribution of Ag core [39]. The weak absorption band at 1008 cm^{-1} is observed because of the C–O stretching of the –COOH group. The absorptions at 1383 cm^{-1} , 1620 cm^{-1} are observed due to C–H deformation and C–C stretching vibrations. The peaks at 2918 cm^{-1} are due to C–H stretching vibrations. A weak absorption band at 3446 cm^{-1} is observed because of the stretching vibration of the O–H functional group, which confirmed the residual citric acid adsorbed onto the surface. Shifts in absorption bands in fig. 4b suggests the successful formation of Ag coated with Fe_2O_3 .

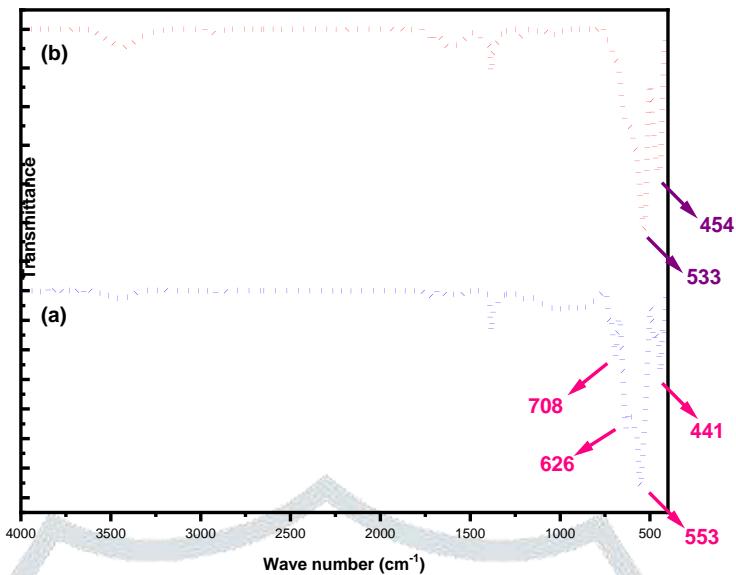


Fig. 4: FTIR spectrum of (a) Fe_2O_3 , and (b) Ag coated with Fe_2O_3

Fig. 5 a demonstrates the FEG-SEM micrograph. The even distribution of Fe and O over Ag indicates the Ag core is evenly coated with a thin layer of Fe_2O_3 shell. Elemental composition/distribution provided by EDAX analysis, as shown in Fig. 5 b. The strong peaks around 3.0 keV and 6.5 keV represent elemental silver and iron as major components of the nanocatalyst. By weight percent composition, Ag and Fe were found to be 51.06% and 29.94 %.

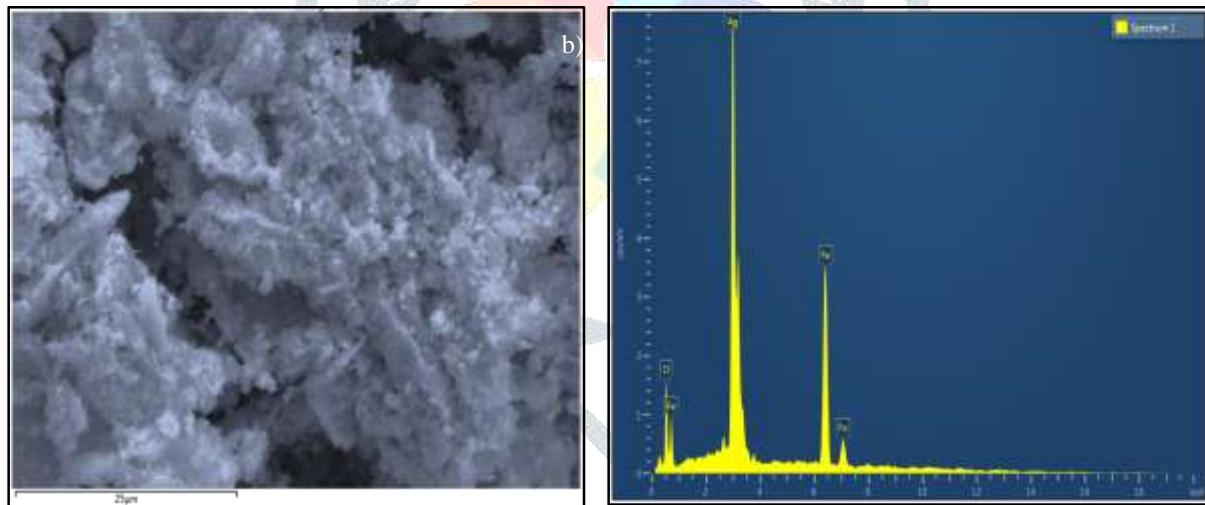


Fig. 5: a) FESEM image; b) EDAX analysis of Ag coated with Fe_2O_3 core-shell nanoparticles

The magnetic nature of as-prepared nanoparticles was studied using a vibrating sample magnetometer. A well-defined hysteresis loop with saturation magnetization (M_s) 28.2 emu g^{-1} and remanence (M_r) 8.68 emu g^{-1} indicates ferromagnetic ordering in the catalyst (fig. 6). Thus, when used as a catalyst, the well-dispersed Ag coated with Fe_2O_3 nanoparticles in the reaction mixture can be easily recovered using an external magnet.

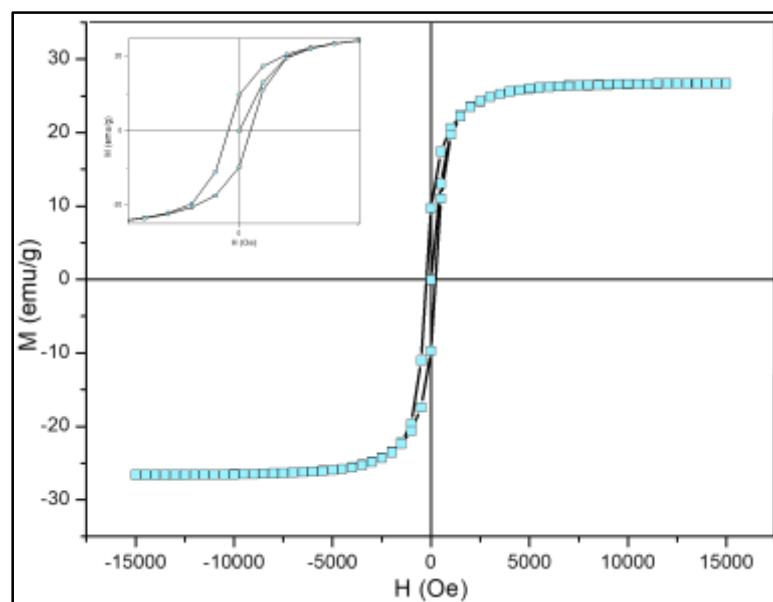


Fig. 6: M-H curve of Ag coated with Fe_2O_3 at R.T. (inset: a) enlarged hysteresis loop; b) magnetically recoverable catalyst)
The thermogravimetric and DSC analysis of nano-Ag coated with Fe_2O_3 was performed between RT and 1000°C at a heating rate of 10°C/min under N_2 purge, which shows no weight loss over the entire range of temperature, which indicates the high thermal stability of the catalyst. The BET surface area of the catalyst was found to be 38.1 m^2/gm and zeta potential -31.4 mV, which shows the colloidal stability of the nanocatalyst in heterogeneous catalytic reactions.

3.2. Synthesis of 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine- 1,8-dione:

A mixture of aromatic aldehyde (1 mmol), dimedone (2 mmol), and ammonium acetate (1 mmol) is heated in the presence of a catalytic amount of Ag coated with Fe_2O_3 (10mg) under solvent-free conditions at Room temperature for the stipulated time. The progress of the reaction was monitored by TLC (n-Hexane: EtOAc, 8:2). The final product was heated in ethanol and poured into water. The catalyst was magnetically removed and washed with chloroform for reuse. The residue was then poured into crushed ice and stirred. The pure product was obtained by recrystallization from hot aqueous alcohol. This resulted in the production of 3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione with a yield of 90-98% (Table 1).

Table 1: Synthesis of 1,8-dioxo-9-aryl decahydroacridine and 1,8-dioxo-9,10-diaryl decahydroacridine derivatives

Entry	Aldehyde	NH ₄ OAc	Time (Min)	Yield ^b (%)	M.P (°C)
1		NH ₄ OAc	10	95	191
2		NH ₄ OAc	08	98	287
3		NH ₄ OAc	12	93	248

4		NH ₄ OAc	15	90	295
5		NH ₄ OAc	11	93	269
6		NH ₄ OAc	09	91	221
7		NH ₄ OAc	19	97	240
8		NH ₄ OAc	15	91	267

^aReaction conditions: aldehyde (1 mmol), dimedone (2 mmol), ammonium acetate (1 mmol) and 10 mg of Ag coated with Fe₂O₃ at room temperature under solvent-free conditions

^bYield of pure isolated products

3.3 Optimization of reaction parameters:

This study presents a novel, simple, gentle, and effective approach for the one-step production of 1,8-dioxodecahydroacridine compounds using Ag coated with Fe₂O₃ as a reusable catalyst. This catalyst exhibits environmentally sustainable properties, possesses secure handling characteristics, and is facile to manage. The initial experiments demonstrated that the reaction of benzaldehyde (1 mmol), dimedone (2 mmol), and ammonium acetate (1 mmol) in the presence of a catalytic amount of Ag coated with Fe₂O₃ (10 mg) under solvent-free conditions at Room temperature for 11 minutes resulted in the production of 3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione with a yield of 95%.

The synthesis of 1,8-dioxodecahydroacridine derivatives was achieved by reacting benzaldehyde, dimedone, and ammonium acetate in different solvents, as indicated in **Table 2**. **Figure 7** depicts the assessment of catalytic loading, indicating that the most favorable output was achieved by utilizing a nanocatalyst of Ag coated with Fe₂O₃ (10 mg) under solvent-free conditions, at a temperature of Room temperature for the model reaction (refer to **Table 1, Entry 1**).

Table 2: Optimization of reaction parameters for 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione derivative synthesis by using Ag coated with Fe₂O₃ catalyst^a (Table 1, Entry 1)

Sr. No	Solvent/Temperature	Time (Min)	Yield ^b (%)
1	THF/ Reflux	44	70
2	DMSO/ Reflux	45	62
3	Ethylene Glycol/ Reflux	40	61
4	Ethanol/ Reflux	30	75

5	H ₂ O/ Reflux	58	40
6	Solvent-free/ Room Temp	10	95
7	Solvent-free/ Reflux	11	95
8	Solvent-free/ 70°C	11	95
9	Solvent free/ 50°C	20	85

^aReaction conditions: benzaldehyde (1 mmol), dimedone (2 mmol), ammonium acetate (1 mmol), and 10 mg of Ag coated with Fe₂O₃

^bYield of pure isolated products

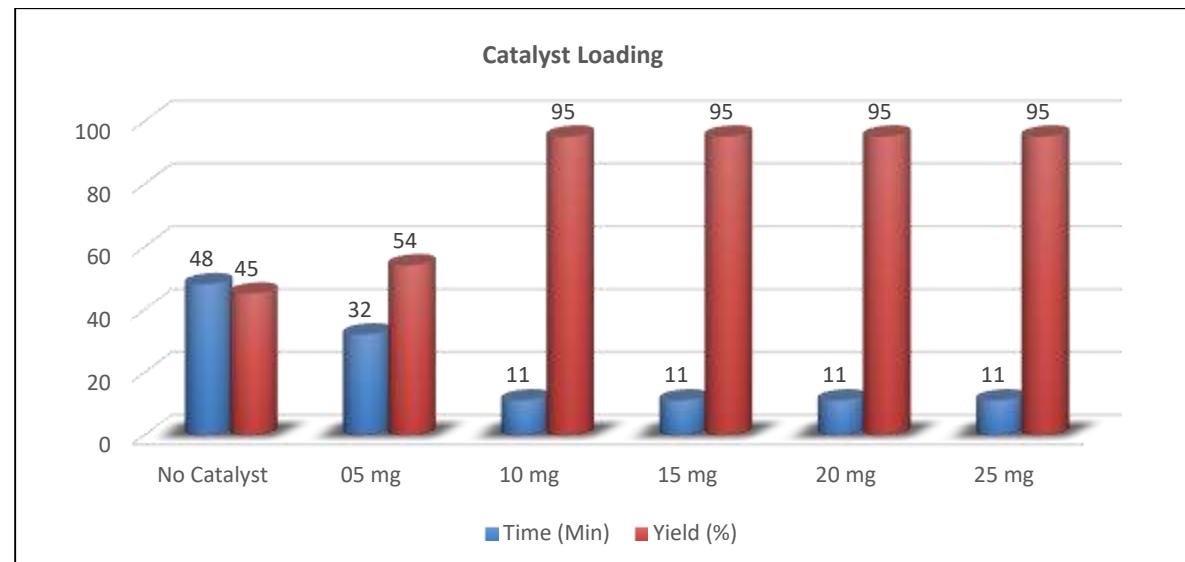


Figure 7: Study of Catalyst loading in the model reaction (Table 1, Entry 1)

To investigate the versatility of the procedure, a variety of aldehydes with different functionalities were reacted in the absence of solvent using dimedone and ammonium acetate along with a catalytic amount of Ag coated with Fe₂O₃ (10mg). The acquired compounds have been methodically organized in a tabular format, as depicted in **Table 1**. The reaction exhibits a high degree of selectivity, as evidenced by the absence of any detectable by-products. The synthesized decahydroacridine1, 8-diones, were characterized through elemental and spectral studies.

3.4 Reusability study:

The Ag coated with Fe₂O₃ nanocatalyst exhibits magnetic recoverability without any significant loss. In order to assess the stability of the catalytic activity and the feasibility of recycling, a series of catalytic cycles were conducted. After each cycle, Ag coated with Fe₂O₃ underwent a chloroform washing process and was subsequently subjected to vacuum drying to eliminate any remaining solvent. The catalyst exhibited reusability for up to five cycles with negligible activity loss, as shown in **Figure 8**. Following each trial, the X-ray diffraction (XRD) pattern of the utilized Ag coated with Fe₂O₃ was found to be indistinguishable from that of the original Ag coated with Fe₂O₃, indicating that the catalyst's framework structure remained intact during the reaction progression. As a result, the catalyst appears to be reusable.

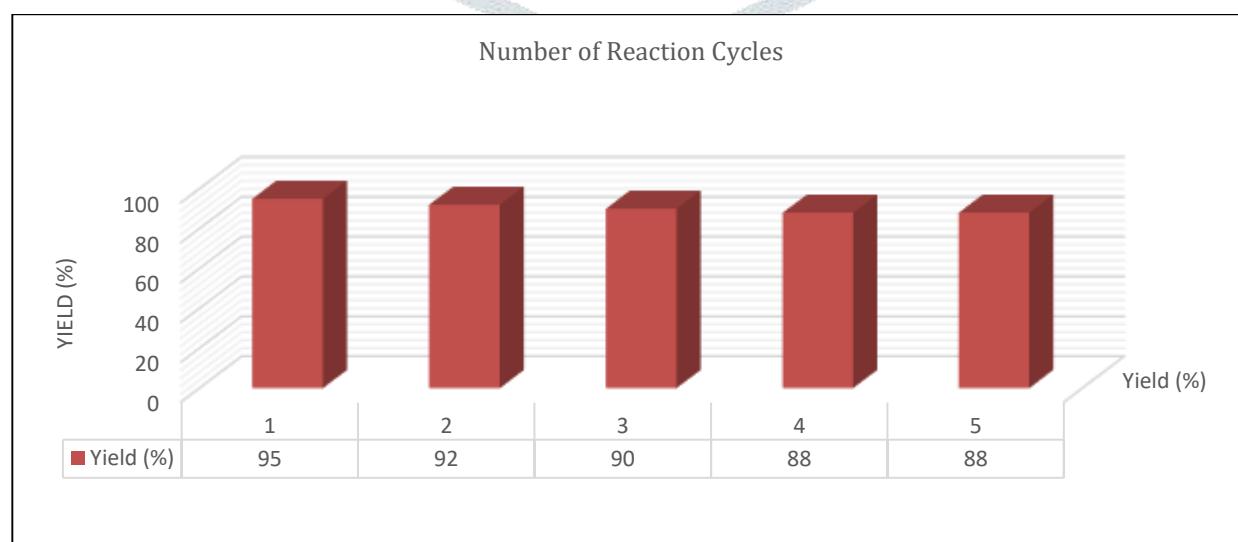


Figure 8: Reusability of the catalyst in the model reaction (Table 1, Entry 1)

Furthermore, the proposed reaction mechanism elucidates the advancement of the multicomponent reaction. At the outset, the catalytic active sites augment the electrophilicity of aldehyde carbonyl groups while facilitating dimedone's enolization. After the activation of the aldehyde, the Knoevenagel product was obtained through a nucleophilic attack of dimedone. Following that, the Knoevenagel product underwent a Michael-type reaction with a second molecule of dimedone, forming an intermediate

compound. Subsequently, the intermediate acquired was subjected to an amine molecule, which underwent intramolecular cyclization through dehydration, forming the ultimate product [23].

3.5. Comparative Study of the Catalyst:

The current investigation explores the catalytic performance of Ag coated with Fe_2O_3 with magnetic properties in the production of a 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione derivative. The comparative analysis of the nanocatalyst's performance was conducted concerning the catalysts reported earlier. **Table 4** demonstrate that the Ag coated with Fe_2O_3 nanocatalyst is an exceptionally potent and proficient catalyst for the synthesis of 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione derivative without the use of any solvents at room temperature. The catalyst outperforms the efficacy of previously documented catalysts. These characteristics make it a highly favorable substance for shell structure.

Table 4: Comparative study for the synthesis of 3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione derivative in the presence of previously reported catalyst^a (Table 1, Entry 1)

Sr. No	Catalyst	Catalyst loading	Solvent	Reaction conditions	Time min	Yield ^b %	Ref No.
1	B6 (FC_5) ₃	3 mol %	solvent-free	RT	3.5 hr	80	[32]
2	TiO_2 NP's	10 mg	solvent-free	80°C	20	90	[23]
3	Silica-Supported Preyssler Nanoparticles	0.03 mmol	H_2O	Reflux	2 hr	91	[33]
4	$\text{Fe}^{3+}/4\text{A}$	0.1 g	EtOH	Reflux	14 hr	91	[34]
5	Ag coated with Fe_2O_3	10 mg	Solvent-free	70°C	11	95	Present work

^aReaction conditions: benzaldehyde (1 mmol), dimedone (2 mmol), ammonium acetate (1 mmol)

^bYield of pure isolated product

IV. CONCLUSION

The study employed the sol-gel auto combustion technique to synthesize Ag coated with Fe_2O_3 core-shell nanoparticles that can be separated magnetically. These nanoparticles were then utilized as a heterogeneous catalyst for the one-pot synthesis of derivatives of 3,3,6,6-tetramethyl-9-aryl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione. Under ambient conditions, a range of aldehydes underwent conversion to their respective acridine-1,8-dione derivatives, with a maximum yield of 98%. The salient characteristics of this investigation encompass the utilization of an environmentally sustainable catalyst, an ecologically sound methodology, a solvent-free facile technique, expeditious reaction kinetics, a substantial product output, and a catalyst that exhibits recyclability without undergoing degradation.

IV. ACKNOWLEDGMENT

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V. CONSENT FOR PUBLICATION

Not applicable

VI. AVAILABILITY OF DATA AND MATERIALS

The authors confirm that the data supporting the findings of this study are available within the article

VII. CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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