One Pot Three Component Synthesis of Substituted 3,4,6,7-Tetrahydro-3,3,6,6-Tetramethyl-9,10-Diphenylacridine-1,8(2H,5H,9H,10H)-Diones Catalyzed by Mesostructured In$_2$O$_3$-SiO$_2$

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ABSTRACT
A convenient, one pot synthesis of heterocyclic nucleous acridine derivative compound achieved those are having very much attention in medicinal chemistry because of their extensive application in biology. The mixture of dimedone, substituted benzaldehyde, and aniline catalyzed by efficient In$_2$O$_3$-SiO$_2$ heterogeneous reusable catalyst in ethanol to obtain the desired acridine derivatives with good yield. The hydrothermal method used to synthesize mesostructured In$_2$O$_3$-SiO$_2$ catalytic material. This catalytic material were characterized by using X-ray diffraction spectroscopy, scanning electron microscopy, energy dispersive spectroscopy, Fourier transform infrared spectroscopy, temperature-programmed desorption, and Brunauer–Emmett–Teller. Our synthetic strategy features high yield, simple work up procedure, nontoxic, clean, and easy recovery and reusability of the catalytic system.

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Mesostructured; In$_2$O$_3$-SiO$_2$; hydrothermal method; acridine

Introduction
The acridine heterocyclic aromatic compound, which entered into the technical and scientific world in 1970, discovered by Grabe and Caro. Due to its pungent smell, this new substance was called “acridin.”$^1$ Ehrlich and Benda first proposed in 1912 that acridine has been used an antimicrobial agent and its clinical used to occur in 1917.$^2$ The tremendous amount of acridine derivatives used in The World War I and II as wound antiseptic. In 1948, acroynoncine extracted from the “Acronychia bauri” Australian tree. The discovery of this a natural acridone alkloide as well as their application in medicinal and pharmaceutical era, to open the opportunity for the young and dynamic researchers to develop the methodology for the preparation of novel acridine analogous those has anticancer and antitumor activity.$^3$–$^6$ Acridines moiety utilized in electronic devices, electroluminescence devices, and light-emitting diodes for this Fadhel and Pretsch file European patent.$^7$

1,8-Dioxodecahydroacridines and their derivatives are poly-functionalized.$^8$–$^9$ Acridine derivatives have been used to synthesize labeled conjugates with medicines, peptides, proteins, and nucleic acids that exhibit antitumor and DNA-binding properties because planer structure of

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acridine. Nowadays, many members of this family used for the treatment of platelet anti-aggregatory activity, Alzheimer’s disease, tumors, cardiovascular diseases as well as hypertension and diabetes.

Literature survey, 1,8-dioxo-decahydroacridines synthesized by various researchers. It reported, 1,3-di (bromo or chloro)-5,5-Dimethylhydantoin, sulfonic acid functionlized silica (SiO$_2$-Pr-SO$_3$H), nano-Fe$_2$O$_3$, HCl, Fe$_3$O$_4$@SiO$_2$–MoO$_3$H nanoparticles, Ru-catalyst, Ionic liquid with multi-SO$_3$H groups like this various catalyst utilized for the synthesis but In$_2$O$_3$-SiO$_2$ combination of binary mixed metal oxide till this date not used for it.

The world is very prompt in the twenty-first century, and no one has enough time. There is a need to customize an efficient heterogeneous catalyst that can pursue multicomponent reactions in one step. In continuation of our studies about to prepared multifunctional mixed metal oxides and utilized in organic transformation. Here, In$_2$O$_3$ is basically considered as basic solid, but when it is doped with other metals its acidity decreases and basicity increases. Reported in literature, there prepared the new design of novel catalytic material, In$_2$O$_3$ is doped with different type metals to synthesized a ternary mixture of metal oxides and utilize for various transformations and gas sensing properties but binary mixture of In$_2$O$_3$-SiO$_2$ as solid heterogeneous catalyst in organic transformation reaction is not studied well till this date. However, crystalline In$_2$O$_3$ combine with mesoporous SiO$_2$ by hydrothermal method to form the multifunctional, cost effective, nontoxic and eco-friendly catalyst. Thus, it is convenient for us, to have less effort because of the simple experimental method for separation and environmentally benign catalyst.

To shed light on the catalytic activity of In$_2$O$_3$-SiO$_2$ of solid heterogeneous catalyst has been tested. Here, we report, a In$_2$O$_3$-SiO$_2$ catalyst utilized for the synthesis of substituted 3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9,10-diphenylacridine-1,8(2H,5H,9H,10H)-diones derivatives using multicomponent reaction between dimedone, substituted benzaldehyde and aniline under reflux condition with ethanol as solvent. The developed protocol has several benefits such as short reaction time, mild reaction condition, reusability of catalyst.

The reactions in the solvent environment had been given very good yield of the final product. Because the liquid phase generates the mobile phase which is helpful for the interaction between reactant and catalyst. These type reactions are simple to handle, reduce pollution, comparatively cheap to operate and are especially important in industry.

**Results and discussion**

At first, to optimize the reaction condition, we studied the reaction of dimedone (2 mmol), benzaldehyde (1 mmol), and aniline (1 mmol) as model compound in the presence of In$_2$O$_3$-SiO$_2$ (1 g) as catalysts in this reaction. We evaluated the effect of different solvents such as CH$_2$Cl$_2$, EtOH, H$_2$O, MeCN, DMF, and solvent-free condition on the reaction rate under the same reaction conditions. EtOH solvent afforded the products in higher yield and shorter reaction time (Table 1).

Reaction Condition: Dimedone (2 mmol), Benzaldehyde (1 mmol), and Aniline (1 mmol) in In$_2$O$_3$-SiO$_2$ catalyst (1 g) EtOH as catalyst; Isolated yields.

| Entry | Solvent   | Time (min) | Yield (%)$^b$ |
|-------|-----------|------------|--------------|
| 1     | Solvent free | 170        | –            |
| 2     | CH$_2$Cl$_2$ | 200        | 43           |
| 3     | H$_2$O     | 230        | 30           |
| 4     | MeCN       | 190        | 75           |
| 5     | DMF        | 240        | 70           |
| 6     | EtOH       | 100        | 92           |

Table 1. Optimization of model reaction (4a) using several solvent.
Table 2. Synthesis of (4a) using different catalysts.

| Entry | Catalyst     | Time (min) | Yield (%) |
|-------|--------------|------------|-----------|
| 1     | In$_2$O$_3$  | 160        | 60        |
| 2     | SiO$_2$      | 190        | 55        |
| 3     | In$_2$O$_3$-SiO$_2$ | 100 | 92        |

Reaction Condition: Dimedone (2 mmol), Benzaldehyde (1 mmol), and Aniline (1 mmol) using In$_2$O$_3$-SiO$_2$ as catalyst; *b* isolated yields.

Table 3. Optimization of amount of catalysis for model reaction (4a).

| Entry | Catalyst (g) | Yield (%) |
|-------|--------------|-----------|
| 1     | 0.3          | 54        |
| 2     | 0.5          | 60        |
| 3     | 0.8          | 88        |
| 4     | 1.0          | 92        |
| 5     | 1.5          | 92        |

Reaction Condition: Dimedone (2 mmol), Benzaldehyde (1 mmol), and Aniline (1 mmol) using In$_2$O$_3$-SiO$_2$ (1 g) as catalyst; *b* isolated yields.

In this study, the effect of different catalyst investigated and shown in (Table 2). When In$_2$O$_3$ and SiO$_2$ powder was employed, the corresponding product yield was obtained 60% and 55%, respectively (Table 2, entry 1, 2). However, the model reaction carried out in In$_2$O$_3$-SiO$_2$ which gave 92% yield. We have found that In$_2$O$_3$-SiO$_2$ as a better catalyst with respective to reaction time and yield of obtained products.

We next investigated the amounts of In$_2$O$_3$-SiO$_2$ catalyst (0.3, 0.5, 0.8, 1.0, 1.5 g). The optimum yield of the 1, 8 dioxo-decahydro acridine was obtained, it was observed that 1 g of In$_2$O$_3$-SiO$_2$ metal oxide was sufficient to drive the reaction completely with 92% yield (Table 3). We found that using less amount of catalyst the yield of reaction product decreases. When more amount of catalyst is used there is no improvement observed in the yield. Therefore, in an optimized reaction condition dimedone (2 mmol), benzaldehyde (1 mmol), aniline (1 mmol) was treated with In$_2$O$_3$-SiO$_2$ (1 g) and reflux at 80 °C for mention time in Table 4.

To study the generality of this process, several examples illustrating this method for the synthesis of those polyfunctionalized 1,8-dioxodecahydroacridines were studied. Results are summarized in (Table 4). The effect of substituents on the aromatic ring did show expected strong effects in terms of yields under these reaction conditions. Aromatic aldehyde derivatives containing electron-withdrawing groups (such as nitro and halo groups) or electron-donating groups (such as alkoxy groups) were employed and they were found to react well to give the corresponding 1,8-dioxodecahydroacridines derivatives in good to excellent yields. Aromatic aldehydes having electron withdrawing groups on the aromatic ring (Table 4, entries 4b–d) react faster than electron-donating groups (Table 4, entry 4e) because the electron withdrawing group helped to generate more electron deficient center on the carbonyl carbon of benzaldehyde, which is easily attacked by enol form of dimedone.

We also examined the recycling performance of In$_2$O$_3$-SiO$_2$ catalyst. It was investigated by using the same model reaction. After the separation of product the catalyst was washed with ethanol for two to three times and dried at 95 °C, and then, used directly with fresh substrates under identical conditions without further purification. It was shown that the catalyst could be used for three runs without a noticeable drop in the product yield and its catalytic activity (Figure 1). The catalyst having easy recycling performance is also an attractive property for the environmental protection and economic reasons.

To explain role of mesosructured In$_2$O$_3$-SiO$_2$ in the mechanism of this tandem Knoevenagel–Michael condensation reaction has been proposed in Figure 2. Initially mesos-structured In$_2$O$_3$-SiO$_2$ attached to keto group of the compound (I) and thereby activate the C═O functional group, which makes it easy for the deprotonation of methylene proton by
In$_2$O$_3$-SiO$_2$ leading to form enolate intermediate Then, the enolate attacks the nucleophilic center of the carbonyl group of aldehyde, followed by Knoevenagel condensation to form Knoevenagel condensation adduct by the loss of H$_2$O molecule. Finally, the second (II) molecule of enolate further attacks the double bond of Knoevenagel product and subsequently undergoes Michael addition reaction to form the tandem Knoevenagel–Michael 1, 5

Table 4. Synthesis of 1, 8 dioxo-decahydro acridine derivatives.

| Entry | Aldehydes | Product | Time (min) | Yield (%)$^b$ | M.P.($^\circ$C) |
|-------|-----------|---------|------------|---------------|----------------|
| 4a    |            | ![Image](image1.png) | 100        | 92            | 210–215, 200–205$^{42}$ |
| 4b    |            | ![Image](image2.png) | 90         | 90            | 240–242, 233–235$^{43}$ |
| 4c    |            | ![Image](image3.png) | 95         | 89            | 235–241, 230–234$^{43}$ |
| 4d    |            | ![Image](image4.png) | 75         | 90            | 278–280, 276–278$^{44}$ |
| 4e    |            | ![Image](image5.png) | 115        | 91            | 285–290, 290–291$^{45}$ |

Reaction condition: Dimedone (2 mmol), Benzaldehyde (1 mmol), and Aniline (1 mmol) in In$_2$O$_3$-SiO$_2$ (1:1) catalyst (1 g) using EtOH as solvent; $^b$Isolated yields.
dicarbonyl compound which is reacted easily with aniline (III) because of generating electrophilic center by mesostructured In$_2$O$_3$-SiO$_2$ to undergo cyclodehydration to form final product 1,8dioxo-decahydroacridine (IV).

**Experimental**

*Synthesis of series of In$_2$O$_3$-SiO$_2$ mixed metal oxide by hydrothermal method*

The In$_2$O$_3$-SiO$_2$ mixed metal oxides were prepared by hydrothermal method. It was prepared from the aqueous solution of InCl$_3$ and tetraethyl orthosilicate (TEOS) in 5 mL of ethanol to taken in separate beakers, respectively. Mixed these two solutions thoroughly in an autoclavable bottle with constant stirring followed by the addition of 1 g cetyl trimethyl ammonium bromide (CTAB) as structural directing agent. This solution was then hydrolyzed by 10 mL 1:1 aqueous ammonia (aq. NH$_3$) with constant stirring until the pH of solution reached to 9–10. At the basic pH the interaction between metal ion and hydroxides is easily carried out helping for complexation. The pale yellowish precipitate formed, was transferred to the teflon beaker in autoclave. This reaction mixture was heating up to 150°C with 400 rpm at 58 psi naturally generated pressure with constant stirring of 24 h. After drying at 110°C for 7 h in an oven, the obtained powder was pulverized using mortar and pestle and finally calcined at 500°C for 2 h. Similarly, all the pure metal oxides and mixed metal oxides prepared by following same procedure. Similarly, by using above same procedure there systematic preparations of pure SiO$_2$, In$_2$O$_3$ and series of different loading In/Si molar ratio is shows in Table 5.

*General procedure for the synthesis of 1,8 dioxo-decahydro acridine 4 (a–e)*

The catalytic activity of In$_2$O$_3$-SiO$_2$ catalyst has been tested for synthesis of 1,8 dioxo-decahydro acridine derivatives by using dimedone (2 mmol), substituted benzaldehyde (1 mmol), and aniline (1 mmol) under reflux condition using ethanol (15 mL) as solvent (Scheme 1). Reaction confirmation is checked by TLC analysis. The solvent was removed at under reduced pressure to obtained acridine product in good and excellent yield. The model reaction product characterized by $^1$H NMR, Fourier transform infrared spectroscopy (FTIR), and Mass spectroscopy. The developed protocol has several benefits such as short reaction time, mild reaction condition, reusability of catalyst.

![Figure 1. The recyclability of In$_2$O$_3$-SiO$_2$ (1:1) for synthesis of 1,8dioxo-decahydroacridine.](image-url)
Spectral data of representative compound

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9,10-diphenylacridine-1,8(2H,5H,9H,10H)-dione (4a)

$^1$H NMR (300 MHz, CDCl$_3$) $\delta = 7.17$–7.27 (m, 5H, Ar-H), 7.08–7.15 (m, 5H, Ar-H), 5.54 (s, 1H, CH), 2.43 (d, 4H, CH$_2$), 2.20 (d, 4H, CH$_2$), 1.09 (s, 6H, 2X CH$_3$), 0.87 (s, 6H, 2X CH$_3$); IR (KBr): 2962(C-H stretch), 1589(C=O), 1450(C=C), 1373(C-N), m/z 426.22 ($M^{+2}$).

Figure 2. Proposed mechanism pathway of 1,8dioxo-decahydroacridine derivatives.
9-(4-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-10-phenylacridine-1,8(2H,5H,9H,10H)-dione (4b)

$^1$H-NMR (CDCl$_3$, 300 MHz): $\delta$ = 7.24 – 7.28 (m, 4H), 7.48 (m, 3H), 7.3 (d, 2H), 5.20 (s, 1H), 2.12 (d, 2H), 2.21 (d, 2H), 1.83 (d, 2H), 1.09 (d, 2H), 1.01 (s, 6H, 2X CH$_3$), 0.82 (s, 6H, 2X CH$_3$); IR (KBr): 2960 cm$^{-1}$ (C-H stretch), 1595 cm$^{-1}$ (C=O), 1495 cm$^{-1}$ (C=C), 1360 cm$^{-1}$ (C-N).

9-(2-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-10-phenylacridine-1,8(2H,5H,9H,10H)-dione (4c)

$^1$H-NMR (CDCl$_3$, 300 MHz): $\delta$ = 7.00 – 7.01 (m, 4H, Ar-H), 6.40 – 7.10 (m, 5H, Ar-H), 4.40 (s, 1H, CH), 2.81 (d, 4H, CH$_2$), 1.80 (d, 4H, CH$_2$), 1.13 (s, 6H, 2X CH$_3$), 0.91 (s, 6H, 2X CH$_3$); IR (KBr): 2933 cm$^{-1}$ (C-H stretch), 1515 cm$^{-1}$ (C=O), 1473 cm$^{-1}$ (C=C), 1347 cm$^{-1}$ (C-N).

3,4,6,7-Tetrahydro-3,3,6,6-tetram ethyl-9-(3-nitrophenl)-10-phenylacridine-1,8(2H,5H,9H,10H)-dione (4d)

$^1$H-NMR (CDCl$_3$, 300 MHz): $\delta$ = 6.40 – 7.10 (m, 5H, Ar-H), 6.51 – 6.97 (m, 4H, Ar-H), 4.41 (s, 1H, CH), 2.84 (d, 4H, CH$_2$), 1.91 (d, 4H, CH$_2$), 1.09 (s, 6H, 2X CH$_3$), 0.82 (s, 6H, 2X CH$_3$); IR (KBr): 2875 cm$^{-1}$ (C-H stretch), 1763 cm$^{-1}$ (electron withdrawing C=O), 1536 cm$^{-1}$ (C=C), 1325 cm$^{-1}$ (C-N).

3,4,6,7-Tetrahydro-9-(4-m ethoxyphenyl)-3,3,6,6-tetram ethyl-10-phenylacridine-1,8(2H,5H,9H,10H)-dione (4e)

$^1$H-NMR (CDCl$_3$, 300 MHz): $\delta$ = 6.47 – 7.09 (m, 5H, Ar-H), 6.61 – 6.97 (m, 4H, Ar-H), 4.39 (s, 1H, CH), 3.15 (s, 3H, CH$_3$), 2.81 (d, 4H, CH$_2$), 1.90 (d, 4H, CH$_2$), 1.27 (s, 6H, 2X CH$_3$), 0.97 (s, 6H, 2X CH$_3$); IR (KBr): 2803 cm$^{-1}$ (C-H stretch), 1740 cm$^{-1}$ (electron donating C=O), 1480 cm$^{-1}$ (C=C), 1220 cm$^{-1}$ (C-N).

Table 5. Synthesis of series of mixed metal oxides by hydrothermal method.

| Entry | Mixed metal oxide | Molar ratio |
|-------|-------------------|-------------|
| 1     | SiO$_2$           | 1           |
| 2     | In$_2$O$_3$       | 1           |
| 3     | In$_2$O$_3$-SiO$_2$ | 0.8:0.2     |
| 4     | In$_2$O$_3$-SiO$_2$ | 0.2:0.8     |
| 5     | In$_2$O$_3$-SiO$_2$ | 1:1         |

Scheme 1. One pot synthesis of 1, 8 dioxo-decahydro acridine derivatives 4(a-e).
Catalyst characterizations

X-ray diffraction analysis

The powder X-ray diffraction (XRD) pattern of SiO₂ and In₂O₃ is shown in Figure 3a,b, respectively. The pure SiO₂ sample consists of a broad diffraction pattern because it is amorphous in nature. The pure In₂O₃ sample exhibit intense and sharp peak due to it is crystalline in nature. The X-ray diffraction patterns of a series of In₂O₃-SiO₂ catalysts calcined at 500°C in the presence of air for 2 h are shown in Figure 3c,d,e. As per results from X-ray powder diffractions, metal oxides and mixed metal oxides samples differ very much indicating incorporation of In₂O₃ into SiO₂ to form In₂O₃-SiO₂ mixed metal oxides.

The XRD pattern of the SiO₂ shows a broad peak at 2θ = 21.74° corresponding to the lattice plane (100) indicates the hexagonal structure of SiO₂. The In₂O₃ X-ray diffraction pattern having intense peaks are observed at 2θ = 21.56°, 30.67°, 35.55°, 51.12°, and 60.77° corresponding to the lattice planes (211), (222), (400), (440), and (622) indicate the cubic structure of In₂O₃. The analysis of a series of In₂O₃-SiO₂ mixed metal oxide it exhibits monoclinic phase could be identified and match with JCPDS card no 76-0152, Here, intense and sharp peaks were obtained at 2θ = 21.44°, 30.52°, 35.40°, 37.64°, 41.79° corresponding to the lattice planes (–101), (–211), (120), (–231), (–320), respectively. XRD pattern data displayed in Figure 3c,d,e.

Consequently, analysis of this XRD pattern simplified by their variation observed in the intensity of peaks due to the content of In₂O₃ and SiO₂. The intensity of the five dominant peaks increases as the content of In₂O₃ enhances in mixed metal oxide Figure 3c, on the other side, as per XRD data in Figure 3d the content of In₂O₃ decreases and SiO₂ increases, then the intensity of these five peaks decreases. Hence, the intense peaks of mixed metal oxides in Figure 3e consist of the same amount of both the oxides and those shown good acidity in the temperature-programmed desorption (NH₃-TPD) analysis. Consequently, such multicomponent reaction acridine derivatives, give good yields of the desired product.

Estimation of particle size using Debye–Scherrer equation

The average crystallite size (T) of different ratio In₂O₃-SiO₂ solid materials can be estimated from X-ray line broadening using the Debye–Scherrer equation.

![Figure 3](image-url). XRD patterns of (a) SiO₂ (b) In₂O₃ and (c) In₂O₃-SiO₂ (0.8–0.2) (d) In₂O₃-SiO₂ (0.2–0.8) (e) In₂O₃-SiO₂ (1–1).
\[ T = \frac{0.94\lambda}{\beta \cos \theta} \]

where \( T \) = average particle size, \( \lambda \) = wavelength, \( \theta \) = diffraction angles, \( \beta \) = FWHM (Full width half maximum). The average crystallite sizes of SiO\(_2\), In\(_2\)O\(_3\), and different ratio In\(_2\)O\(_3\)-SiO\(_2\) materials are summarized in Table 6.

**FTIR analysis**

Figure 4a shows the FTIR spectrum of the synthesized SiO\(_2\) material. The absorption band at 3400 cm\(^{-1}\) is due to the Si-OH is stretching vibration, 2337 cm\(^{-1}\) for glass SiO\(_2\), 1527 cm\(^{-1}\) for the Si-OH bending mode, 1095 cm\(^{-1}\) for Si-O stretching vibration and 802 cm\(^{-1}\) due to the Si-O-Si bending vibration mode. Figure 4b shows the FTIR spectrum of In\(_2\)O\(_3\) metal oxide having an absorption band at 3390 cm\(^{-1}\) which is due to the In-OH is stretching vibration. The strong absorption band at 493 cm\(^{-1}\) is due to the antisymmetric In–O–In vibrational mode of In\(_2\)O\(_3\) metal oxide. Figure 4c shows the FTIR spectra of the In\(_2\)O\(_3\)-SiO\(_2\) materials having absorption band at 3410 cm\(^{-1}\) due to the Si-OH stretching vibration, 1689 cm\(^{-1}\) due to the Si-OH bending mode, 1087 cm\(^{-1}\) for Si-O stretching vibration and 810 cm\(^{-1}\) due to the Si-O-Si bending vibrational mode the strong absorption band at 462 cm\(^{-1}\) is due to the antisymmetric In–O–In vibrational mode of In\(_2\)O\(_3\).\(^{47-48}\)

**SEM-EDS analysis**

The scanning electron microscopy (SEM) produces artificial three-dimensional point by point reconstruction of the sample from a signal emitted when the electron beam interacts with the specimen. When the beam interacts with the sample, several sorts of signals are produced two basic signals are electron and electromagnetic radiation. If SEM is operated so as to produce an image using backscattered electrons, it should be noted that resolution is considerably reduced in the back scattered mode of operation, because back scattered electrons are generated in a large volume of sample (1–3 nm deep). In this study, it is necessary to consider not only intrinsic instrument resolution but also the interaction of the incident electron beam and the sample. SEM and energy dispersive spectroscopy (EDS) measurements were applied to analyze the detailed morphology, surface roughness, and structure of the material alongside element detection.\(^{50}\)

Figure 5a shows the flakes like structure of SiO\(_2\) and Figure 5b indicate the surface morphology of In\(_2\)O\(_3\) oxide shows grain like structure. Figure 5c indicates clearly deposition of In\(_2\)O\(_3\) on the surface of the SiO\(_2\) metal oxide.

Elemental compositions of In\(_2\)O\(_3\)-SiO\(_2\) metal oxide (Figure 6), were represented in the EDS spectrum. The In, Si, and O peaks can be obviously found in spectrum indicate that the In\(_2\)O\(_3\)-SiO\(_2\) binary metal oxide particles were successfully prepared and there was complete removal of capping agent. The mass % of In, Si, O was found 2.75, 46.74, 50.51 shown in Table 7 and confirmed the presence of In, Si, and O with corresponding empirical formula is In\(_1\) Si\(_{17}\) O\(_{18}\).
Temperature-programmed desorption (NH$_3$-TPD)

NH$_3$-TPD measurements were carried out by analytical instrument MICROMERITICS CHEMISORB 2750 TPD/TPR. Temperature programmed desorption (TPD) studies were done using 100 mg of the sample loaded on a quartz reactor. The samples were first treated with 150°C in helium flow 25 cc/min for 1 h at room temperature. Desorption was carried out at a heating rate of 10°C/min. Temperature programmed desorption of ammonia serves as a dependable technique for the quantitative determination of the acid strength distribution. After cooling
to room temperature, ammonia was injected in the absence of the carrier gas flow and the system was allowed to attain equilibrium. A current of nitrogen was used to flush out the excess and physic-sorbed ammonia. The temperature was then raised in a stepwise manner at a linear heating rate of about 10 °C/min. The ammonia desorbed from 100 °C to 500 °C.

Figure 6. EDS spectrum of In$_2$O$_3$-SiO$_2$ (1:1).

Table 7. EDS elemental quantitative analysis of In$_2$O$_3$-SiO$_2$ (1:1).

| Constituents | Mass (%) | Atom (%) |
|--------------|----------|----------|
| In           | 12.97    | 2.75     |
| Si           | 53.87    | 46.74    |
| O            | 33.17    | 50.51    |
| Total        | 100.00   | 100.00   |

Figure 7. NH$_3$-TPD profiles of In$_2$O$_3$-SiO$_2$ (1:1).
NH$_3$-TPD method was used to determine the acidic properties of solid catalyst. This provides information about the total concentration and strength of acidic sites (Bronsted and Lewis). From TPD analysis data, it was found that the ammonia desorbed in region volume of 0.7236 mmol/gm of NH$_3$ desorbed at 190.38 °C, it indicates that In$_2$O$_3$-SiO$_2$ (1:1) possesses weak acidic sites 0.02957 mmol/gm (Figure 7).

BET analysis

Brunauer–Emmett–Teller (BET) surface area was measured by means of N$_2$ adsorption at 77.74 K preformed on a Micromeritics, ASAP 2010. BET theory aims to explain the physical adsorption of gas molecules on a solid surface and proved as an important analysis technique for the measurement of the specific surface area of a material is the so-called BET method.
(Brunauer–Emmett–Teller). \( \text{N}_2 \) adsorption-desorption isotherms provide information on the textural properties of In\( _2 \)O\( _3 \)-SiO\( _2 \) (1:1) and the specific surface area. The specific surface area and average pore diameter of In\( _2 \)O\( _3 \)-SiO\( _2 \) (1:1) are to be found 62.6013 m\(^2\)/g and 8.82 nm Figures 8 and 9.

**Conclusion**

In summary, Due to the combination of In and Si metals to form the efficient and cost effective catalyst has been developed for the synthesis of acridine derivatives. We take the help of XRD, SEM, EDS, BET, and FTIR characterization technique’s for the analysis of surface morphology, shape, elemental composition, particle size, and structure, respectively, of In\( _2 \)O\( _3 \)-SiO\( _2 \) catalytic material. We have developed a straight forward and efficient methodology for synthesis 1, 8 dioxo-decahydro acridine derivatives using dimedone, substituted benzaldehyde, and aniline under reflux condition using In\( _2 \)O\( _3 \)-SiO\( _2 \) catalyst in ethanol as solvent. The In\( _2 \)O\( _3 \)-SiO\( _2 \) catalyst is high thermal stability and reusability without any noticeable loss of reactivity. The mild reaction conditions, high catalytic activity, and simplicity of the procedure offers several advantages such as nontoxic, noncorrosive, shorter reaction time, simple experimental procedure, high yield, and catalyst can be recycled and reused.

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**Disclosure statement**

No potential conflict of interest was reported by the authors.

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