



One pot synthesis of benzopyranones and benzoxazinones catalyzed by MMO

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One pot synthesis of benzopyranones and benzoxazinones is aimed to develop an environment friendly strategy in which the requisite moiety is constructed by using functionality of chalcones and mixed metal oxides (MMO). MMO, as catalyst are the topic of continuing interest due to their versatile applications in the field of organic synthesis. A novel approach to the synthesis of benzopyranones and benzoxazines with high pharmaceutical activity is sought to eliminate the use of volatile organic solvents. A new methodology is designed to carry out the synthesis in a single step with enhance reaction rate and high yield.

Keywords: MMO (Mixed metal oxide), Benzopyranones, Benzoxazinones (BOZ), Nucleophiles, Heterocondensation

The environment calls on the entire research edifice to define long term goals for clean chemistry and to reduce the dependence on ecologically unsafe chemicals, it is advantageous to carry out reactions in green methodology. Coumarin or benzopyran-2[H]-one is a medically important heterocyclic scaffold used in various synthetic purposes¹⁻⁵. Fusion of coumarin to pyrimidine / isoxazoles/ pyrazol rings to form polycyclic fused compounds may result in enhanced pharmaceutical applications efficiently. On the other hand, Benzoxazinone derivative is a non-nucleoside reverse transcriptase inhibitor and used in clinical treatment of AIDS⁶⁻⁹. To derive more efficacious drug, this moiety is always in considerable attention^{10,11}. Motivated by the aforementioned findings and in continuation of our studies on MMO as catalyst, it was thought worthwhile to design the synthesis of benzopyranones and benzoxazinones that can be envisaged as a new route. In the present communication, aiming at the preparation of Dihydrobenzo[1,3]oxazin-4-ones and Dihydrochromanel [4,3] isoxazol/ pyrimidine / thioxo-4-ones, we investigated for the first time a one-step condensation of *o*-hydroxyaromatic aldehyde, hydroxyl amine hydrochloride, and various aromatic and hetero-aromatic aldehydes catalyzed by Ca-Al mixed metal oxide (CaAl₂O₄). 3-arylidene chromane-2, 4-diones condenses with the MMO along with three different nucleophiles to get the desired product. Moreover versatility of different other MMOs was also explored for this synthetic approach but Ca-Al mixed metal

oxide was found to be best. Nano sized metal oxides are highly active for a large number of reactions that are important both in pollution control and chemical synthesis. In recent years mixed metal oxides (MMO) are being used as solid catalysts either as active phases or as supports¹². Bulk MMOs have proven applications in the areas of catalysis and technology because of their versatile chemical and physical properties especially their acid-base and redox catalytic properties¹³⁻¹⁸. Nanomaterials exhibit novel characteristics compared to bulk material due to their special structural variations. The size, surface structure and inter-particle interaction of nanoparticles lead to unique properties and therefore they have great potential to act as excellent catalysts¹⁹. Thus, the development of an efficient methodology using MMO as catalyst appears to be attractive. One pot synthesis of benzopyranones and benzoxazinones with MMO as catalyst, resulting in excellent yields in lesser reaction time may prove to be a paradigm shift away from using conventional catalysts.

Experimental

The temperature of the reaction mixture was measured with a non-contact minigun type IR thermometer (model 8868). IR spectra were recorded with a Perkin-Elmer FTIR -1710 spectrometer using KBr pellets. ¹H NMR spectra were obtained with a Bruker Avance - Spectrospin 300 spectrometer (300MHz) using TMS as internal standard. Elemental analyses were performed with Heraeus CHN-Rapid

analyzer. The melting points (uncorrected) were determined with a Thomas Hoover melting point apparatus. Mass spectra were recorded with a TOF-MS instrument. All reactants were purchased from Sigma-Aldrich and Lancaster and used without further purification. Solvents used for the reactions were double distilled in a vacuum. Mixed metal oxides (CaAl_2O_4) was synthesized by co-precipitation method. The synthesized MMO was characterized by various analytical techniques such as SEM, TEM, and XRD in order to obtain structural information and optimization of calcination temperature of the product. D Bruker – D8 Discover X-ray diffractometer with Cu - $K\alpha$ radiation generated at 40 kV and 40 mA, was used to analyze X-Ray diffraction patterns for samples. The XRD analysis of the prepared catalyst was carried out to characterize the mixed metal oxides formed. Powder XRD patterns of (CaAl_2O_4) shows it to be cubic structure. The surface morphology and EDAX was monitored using Scanning Electron Microscope (Model 6610LV, JEOL, and Tokyo, Japan). The EDAX data supported the characterization of mixed metal oxides. To gain insight of structure and particle size transmission electron microscopy was employed (model TECNAI GT30). Some of the individual particles are well resolved and their sizes are found to be less than 30 nm in TEM images but in general these particles tend to agglomerate as is evident from TEM images (Fig. 1). The TEM images obtained for the given sample reveals that the sample is having uniform distribution of crystallite size about 20 nm.

Synthesis of dihydrochromanel[4,3] isoxazol (5a-c)/ pyrimidine (5d-f) / thioxo-4-ones (5g-i)

4-hydroxycoumarin **1** (0.01 mol.) and aromatic aldehyde or hetero aromatic aldehyde **2** were taken in round bottom flask followed by addition of 10 mL ethanol and 6M solution of NaOH, the reaction mixture was stirred for 30 min, the progress of reaction was monitored by TLC with mean particle size 10-12 μm , particle distribution 20 μm with solvent system [ethyl acetate: benzene 2:8 (v/v)]. After 30 min of heating, ice cold water containing 10% hydrochloric acid was added to the reaction mixture until the precipitation completed. The solid obtained was filtered and washed with water. To the obtained solid 3-arylidenechromane-2,4-diones **3** (0.01 mol.), a nucleophile, hydroxyl amine hydrochloride **4a** / urea **4b**/ thiourea **4c** (0.015 mol) and Ca-Al mixed metal oxide was added. The reaction mixture was refluxed

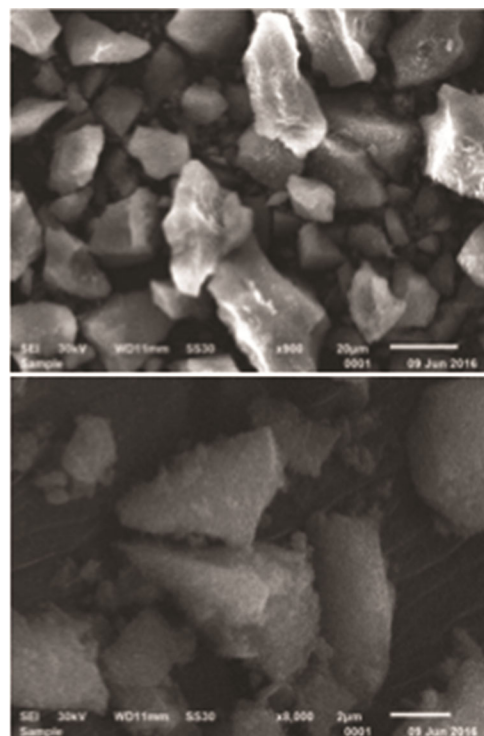
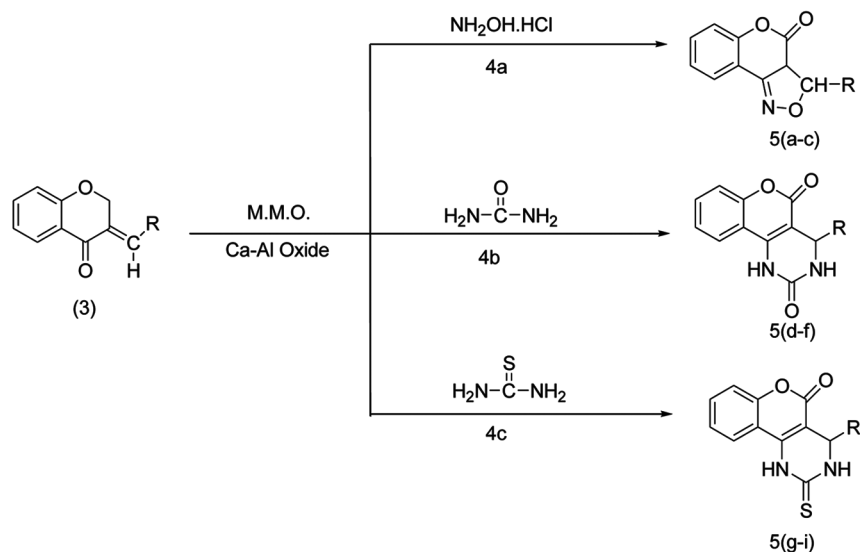


Fig. 1 — Transmission electron micrograph

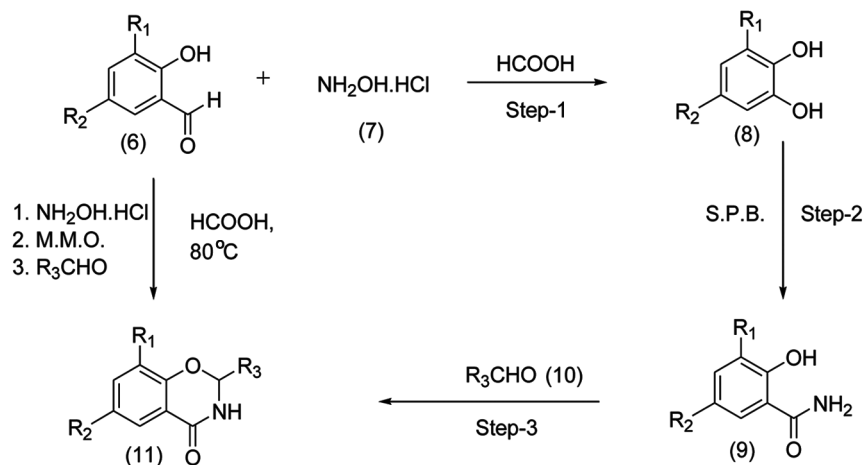
for ~30 mins with constant stirring. The progress of reaction was monitored by thin layer chromatography. Upon completion of the reaction, the solid obtained was filtered and washed with water. Then the product **5a**, **5b**, **5c** was purified by column chromatography [silica gel, elution with benzene and ethyl acetate in 8:2(v/v)] and followed by recrystallisation using ethanol (Scheme 1).

Synthesis of 2-substituted-2,3-dihydrobenzo [1,3] oxazin-4-ones, **11**

A solution of hydroxyl aromatic aldehyde **6** (0.01 mol) and hydroxyl amine hydrochloride **7** (0.013 mol) was taken in 99% formic acid (5-10 mL) which was then refluxed for 1-2 h and 0.005 g of mixed metal oxide (Ca-Al oxide) was added. The mixture was refluxed and stirred for 6-7 h at 80°C. The progress of the reaction was monitored through TLC with mean particle size 10-12 μm , particle distribution 20 μm and aldehyde (0.01 mol) was added. The reaction mixture was allowed to cool down and filtered. Reaction mixture was then diluted and neutralized using 50-60 mL of 5% sodium hydroxide and cooled in ice bath and extracted using ether (2×30 mL). The ethereal layer was extracted and was dried using sodium sulphate and concentrated by rotary evaporator to give final product **11** which is



Scheme 1 — General procedure for the synthesis of dihydrochroman[4,3]isoxazol (5a-c)/ pyrimidine (5d-f) / thioxo-4-ones (5g-i)



Scheme 2 — Synthesis of 2-substituted-2,3-dihydrobenzo [1,3] oxazin-4-ones 11

further purified using column chromatography [silica gel, elution with benzene and ethyl acetate in 8:2(v/v)] and followed by recrystallisation using ethanol (Scheme 2).

Spectral characterization data

3-Phenyl-3,3a-dihydrochromeno[4, 3-c]isoxazol-4-one, 5a: m. p. 167°C (Lit. $167\text{-}168^\circ\text{C}^{29a}$). Anal. Calcd for $\text{C}_{16}\text{H}_{11}\text{O}_3\text{N}$: C, 72.45; H, 4.15; N, 5.28. Found: C, 72.35; H, 4.18; N, 5.30%. IR (nujol): $1615, 1670\text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3 TMS): δ 2.25 (d, 1H, $J = 9.8$ Hz), 5.50 (d, 1 H, CHO, $J = 8.0$ Hz), 6.81 -7.66 (m, 9H, Ar-H). MS: m/z (%) 270.8 (100) $[\text{M}]^+$.

3-(4-Methoxyphenyl)-3,3a-dihydro-chromenol [4, 3-c]-isoxazol-4-one, 5b: m. p. 190°C (Lit. 188 -

190°C^{29a}). Anal. Calcd for $\text{C}_{17}\text{H}_{13}\text{O}_4\text{N}$: C, 69.15; H, 4.40; N, 54.74. Found: C, 69.13; H, 4.38; N, 4.72%. IR (nujol): $1605, 1660\text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3 TMS): δ 2.27 (d, 1H, $J = 9.8$ Hz), 3.84 (s, 3 H, OCH₃), 5.50 (d, 1 H, CHO, $J = 8.0$ Hz), 6.86 -7.53 (m, 8H, Ar-H); $^{13}\text{C NMR}$ (75.6 MHz, CDCl_3 , TMS): δ 129.6, 125.8, 133.2, 123.5, 154.6, 171.0, 47.3, 66.8, 128.5, 129.9, 134.3, 129.5, 128.6, 139.0, 166.6, 125.1, 61.2. MS: m/z (%) 295.2 (100) $[\text{M}]^+$.

3-(4-Chlorophenyl)-3,3a-dihydrochromenol[4,3-c]isoxazol-4-one, 5c: m. p. 176°C (Lit. $175\text{-}177^\circ\text{C}^{29a}$). Anal. Calcd for $\text{C}_{16}\text{H}_{10}\text{O}_3\text{NCl}$: C, 64.10; H, 3.33; N, 4.67. Found: C, 64.13; H, 3.34; N, 4.68%. IR (nujol): $1615, 1665\text{ cm}^{-1}$; $^1\text{H NMR}$ (300 MHz, CDCl_3 TMS): δ 2.25 (d, 1H, $J = 9.8\text{ Hz}$), 5.59 (d, 1 H, CHO $J = 8.0$

Hz), 6.80-7.55 (m, 8H, Ar-H). MS: m/z (%) 299.3 (100) $[M]^+$.

4-Phenyl-1,2,3,4-tetrahydrobenzopyranol[4,3-d]pyrimidine-2,5-dione, 5d: m. p. 162-163°C (Lit. 162°C^{29b}). Anal. Calcd for C₁₇H₁₂O₃N₂: C, 69.86; H, 4.10; N, 9.58. Found: C, 69.85; H, 4.14; N, 5.30%. IR (nujol): 1680, 3445 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 6.12 (s, 1H), 6.80-7.40 (m, 9H, Ar-H) 11.53 (brs, 2H, 2NH). MS: m/z (%) 292.0 (100) $[M]^+$.

4-(4-Methoxyphenyl)-1,2,3,4-tetrahydrobenzopyranol[4,3]pyrimidine-2,5-dione, 5e: m. p. 240 - 242°C (Lit. 240°C^{29b}). Anal. Calcd for C₁₈H₁₄O₄N₂: C, 67.08; H, 4.34; N, 8.69. Found: C, 67.05; H, 4.31; N, 8.73%. IR (nujol): 1660, 3455 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 6.10 (s, 1H), 3.86 (s, 3H, OCH₃), 6.80-7.40 (m, 8H, Ar-H) 11.53 (brs, 2H, 2NH). MS: m/z (%) 321.8 (100) $[M]^+$.

4-(4-Chlorophenyl)-1,2,3,4-tetrahydrobenzopyranol[4,3]pyrimidine-2,5-dione, 5f: m. p. 161-163°C (Lit. 162-164°C^{29c}). Anal. Calcd for C₁₇H₁₁O₃N₂Cl: C, 62.48; H, 3.36; N, 8.57. Found: C, 62.48; H, 3.36; N, 8.54%. IR (nujol): 1672, 3450 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 6.10 (s, 1H), 6.80-7.40 (m, 8H, Ar-H) 11.52 (brs, 2H, 2NH). MS: m/z (%) 326.0 (100) $[M]^+$.

4-Phenyl-1,2,3,4-tetrahydrobenzopyranol[4,3]pyrimidine-2-thioxo-5-one, 5g: m. p. 188-190°C (Lit. 188°C^{29b}). Anal. Calcd for C₁₇H₁₂O₂N₂S: C, 62.23; H, 3.89; N, 9.09, S 10.38. Found: C, 62.22; H, 3.87; N, 9.07, S 10.39%. IR (nujol): 1670, 3445 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 6.10 (s, 1H), 6.80-7.40 (m, 9H, Ar-H) 11.48 (brs, 2H, 2NH); ¹³C NMR (75.6 MHz, CDCl₃, TMS): δ 128.6, 125.0, 130.0, 122.3, 152.8, 171.0, 101.9, 58.2, 129.1, 130.2, 128.3, 130.0, 128.7, 144.4, 184.2, 163.2, 129.7. MS: m/z (%) 308.0 (100) $[M]^+$.

4-(4-Methoxyphenyl)-1,2,3,4-tetrahydrobenzopyranol[4,3-d]pyrimidine-2-thioxo-5-one, 5h: m. p. 234-236°C (Lit. 234°C^{29b}). Anal. Calcd for C₁₈H₁₄O₃N₂S: C, 63.85; H, 4.14; N, 8.28; S, 9.46. Found: C, 63.89; H, 4.12; N, 8.25; S, 9.45%. IR (nujol): 1672, 3450 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ =6.10 (s, 1H), 3.84 (s, 3H, OCH₃), 6.07 (s, 1H), 6.80-7.40 (m, 8H, Ar-H), 11.48 (brs, 2H, 2NH). MS: m/z (%) 338.6 (100) $[M]^+$.

4-(4-Chlorophenyl)-1,2,3,4-tetrahydrobenzopyranol[4,3-d]pyrimidine-2-thioxo-5-one, 5i: m. p. 187-189°C (Lit. 188-190°C^{29c}). Anal. Calcd for C₁₇H₁₁O₂N₂ClS: C, 59.56; H, 3.21; N, 8.17; S, 9.34. Found: C, 59.54; H, 3.20; N, 8.1; S, 9.35%. IR

(nujol): 1680, 3450 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 6.08 (s, 1H), 6.80-7.82 (m, 8H, Ar-H), 11.48 (brs, 2H, 2NH). MS: m/z (%) 342.0 (100) $[M]^+$.

2-Phenyl-2,3-dihydrobenzo[e][1,3]oxazin-4-one, 11a: m. p. 166-168°C (Lit. 168°C^{29d}). Anal. Calcd for C₁₆H₁₁O₃: C, 74.66; H, 4.88; N, 6.22. Found: C, 74.65; H, 4.86; N, 6.23%. IR (nujol): 3160, 3060, 1675 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ =7.03(s, 1 H), 7.30-8.20 (m, 9H, Ar-H), 10.70(s, 1H, N-H). MS: m/z (%) 225.0 (100) $[M]^+$.

2-(2-Hydroxyphenyl)-2,3-dihydrobenzo[e][1,3]oxazin-4-one, 11b: m.p. 171°C (Lit. 168°C^{29e}). Anal. Calcd for C₁₄H₁₁O₃N: C, 69.70; H, 4.56; N, 5.80. Found: C, 69.66; H, 4.59; N, 5.84%. IR (nujol): 3350, 3160, 3065, 1670 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ =7.15(s, 1 H), 7.24-8.10 (m, 8H, Ar-H), 10.70(s, 1H, N-H). MS: m/z (%) 270.8 (100) $[M]^+$.

2-(4-Chlorophenyl)-2,3-dihydrobenzo[e][1,3]oxazin-4-one, 11c: m. p. 204-205°C (Lit. 205-206°C^{29f}). Anal. Calcd for C₁₄H₁₀O₂Cl: C, 64.73; H, 3.85; N, 5.39. Found: C, 64.75; H, 3.85; N, 5.36%. IR (nujol): 3165, 3060, 1680 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 7.10(s, 1 H), 7.24(d, 2H, Ar-H), 7.40 (d, 2H, Ar-H), 7.50-8.20 (m, 4H, Ar-H), 10.70(s, 1H, N-H). MS: m/z (%) 259.8 (100) $[M]^+$.

2-(3-Nitrophenyl)-2,3-dihydrobenzo[e][1,3]oxazin-4-one, 11d: m. p. 217-218°C (Lit. 217-218°C^{29f}). Anal. Calcd for C₁₄H₁₀O₄N₂: C, 62.22; H, 3.70; N, 10.37. Found: C, 62.21; H, 3.69; N, 10.37%. IR (nujol): 3170, 3055, 1680 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ =7.15(s, 1 H), 8.20 (s, 1H, Ar-H), 7.50-8.10 (m, 7H, Ar-H), 10.70(s, 1H, N-H); ¹³C NMR (75.6 MHz, CDCl₃, TMS): δ 99.09, 161.59, 119.27, 129.61, 120.33, 132.89, 114.44, 158.59, 143.49, 122.27, 149.28, 121.67, 129.67, 134.07. MS: m/z (%) 270.8 (100) $[M]^+$.

2-(4-Methoxyphenyl)-2,3-dihydrobenzo[e][1,3]oxazin-4-one, 11e: m. p. 165-166°C (Lit. 166-167°C^{29f}). Anal. Calcd for C₁₅H₁₃O₃N: C, 70.58; H, 5.09; N, 5.48. Found: C, 70.56; H, 5.07; N, 5.51%. IR (nujol): 3175, 3065, 1675 cm⁻¹; ¹H NMR (300 MHz, CDCl₃ TMS): δ 3.84 (s, 3H, OCH₃), 7.00(s, 1 H), 7.20 (d, 2H, Ar-H), 7.40(d, 2H, Ar-H), 7.50-8.10 (m, 4H, Ar-H), 10.70(s, 1H, N-H). MS: m/z (%) 255.3 (100) $[M]^+$.

Results and Discussion

Numerous methods reported for preparation of various derivatives of benzopyranones so far employ the use of hazardous solvents like

diphenylnitrilimine²⁰, acetic acid²¹, pyridine²², xylene²³ and harmful catalysts like triethylamine²⁴ and for synthesis of ben-zoxazinones, triphenylphosphine²⁵ acetic anhydride²⁶, and bisphenols²⁷, *etc.*²⁸ are used. The procedures for the desired organic functional group transformation were made green by using MMO as catalysts as explained in Schemes 1 and 2.

Initially 4-hydroxy-2benzopyran-2-one (**1**) was condensed with an aromatic or hetero aromatic aldehyde (**2**) in ethanol and sodium hydroxide to yield 3-arylidene chromane-2,4-diones (**3**). The reaction time to yield intermediate (**3**) was 30 mins which was easily isolated in the same pot Ca-Al mixed metal oxide along with three other nucleophiles (hydroxyl amine hydro chloride **4a**/urea **4b** / thiourea **4c**) were added to the pot to obtain the final products as 3-substituted aryl,3,3a-dihydrochromane[4,3]isoxazol-4-ones (**5a-c**)/4-substituted-1,2,3,4-tetrahydrobenzopyranol [4,3]pyrimidene-2,5-diones (**5d-f**)/4-substituted-1,2,3,4-tetrahydrobenzopyranol-[4,3]pyrimidene-2,thioxo-5-ones (**5g-i**) (Table 1). Different solvents such ethanol, water, benzene in different combinations were studied to achieve atom economy within shorter reaction time (Table 2). The cyclisation after forming chalcone that took almost 30mins gave derivatives after stirring reaction mixture for 4-5 h. The addition of MMO served as catalyst in the schemes to yield derivative using hydroxyl amine hydrochloride, urea and thiourea which were soluble and thus nucleophiles react further to obtain final products as **5a-c**, **5d-f** and **5g-i**.

In the case of BOZ derivatives, according to literature survey, synthesis of 2-substituted-2,3-

dihydrobenzo[1,3]oxazin-4-one has been carried out in three different steps where in first step *o*-hydroxybenzoxazole **6** was prepared consisting two functional groups (-CN and -OH), in the presence of various oxidizing agents to yield *o*-hydroxybenzamide intermediate **9**, which undergoes cyclisation reaction with aldehydes **10** to give 2-substituted-2,3-dihydrobenzo[1,3]oxazin-4-one as product **11**.

By optimizing the reaction conditions to obtain required product, the 3-step reaction was successfully conducted in one pot heterocyclic reaction of *o*-hydroxy aromatic aldehyde **6**, hydroxyl amine hydrochloride **7** and various aromatic and heteroaromatic aldehydes **10** catalyzed using Ca-Al mixed metal oxide as shown below in Scheme 1. *o*-Hydroxyaldehyde **6** was taken in hydroxyl amine hydrochloride **7** in presence of formic acid where *in situ* formed oxime gets transformed to cyanide through elimination of water to give *o*-hydroxybenzoxazole **8** without involving -OH group. The progress of reaction was checked using TLC, followed by addition of Ca-Al mixed metal oxide that specifically hydrolyses cyano group **8** to amide here again -OH group is untreated and leads to formation of *o*-hydroxy benzamide **9** intermediate in the reaction mixture and further addition of aromatic and heteroaromatic aldehydes **10** leads to the formation of final product **11**. The reaction in different solvent system other than formic acid like chloroform, ethyl acetate, was tried. But poor solubility of both hydroxylamine hydrochloride and MMO in these solvents led to lower yield. The structure of synthesized product was obtained with confirmed spectroscopic and elemental analysis.

Table 1 — Synthesis of 3-substituted aryl,3,3a-dihydrochromane[4,3]isoxazol-4-ones (**5a-c**)/4-substituted-1,2,3,4-tetrahydrobenzopyranol[4,3]pyrimidene-2,5-diones(**5d-f**)/4-substituted-1,2,3,4-tetrahydrobenzopyranol[4,3]pyrimidene-2,thioxo-5-ones (**5g-i**)

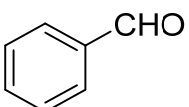
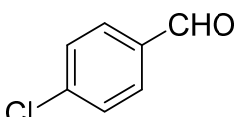
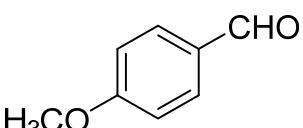
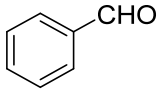
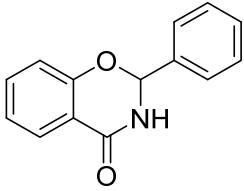
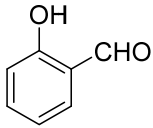
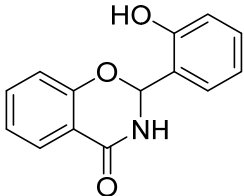
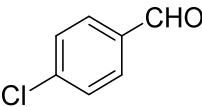
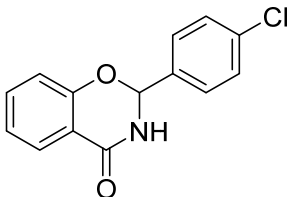
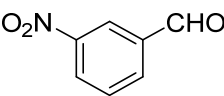
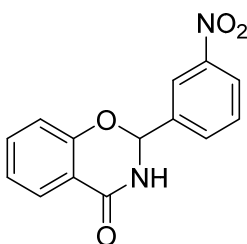
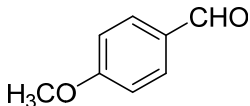
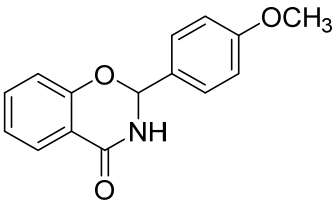
S. No.	Product	R	Yield (%)	Time (h)
1.	5a 5d 5g		87	6-7
2	5b 5e 5h		90	4-5
3	5c 5f 5i		89	5-6

Table 2 — Study of atom economy and reaction time

S. No.	R ₁	R ₂	R ₃	Product 11a-e	Yield (%)	Time (h)
1.	H	H			85	7-8
2.	H	H			75	7-8
3.	H	H			88	6-7
4.	H	H			85	7-8
5.	H	H			92	7-8

Conclusion

In conclusion a synthetic route to various fused heterocycles catalysed by MMO was designed without using any harsh acids or bases thereby eliminating the use of toxic compounds and solvents. Moreover the use of MMO resulted in complete conversion of reactants into products without the formation of side products and any noticeable decomposition.

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