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Research Paper

Chitosan Catalyzed One-Pot Three-Component Conventional Synthesis of 1,2-Dihydro-1-Arylnaphtho[1,2-*e*][1,3]Oxazine-3-Ones

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ABSTRACT

A series of 1,2-dihydro-1-arylnaphtho[1,2-*e*] [1,3] oxazine-3-ones was synthesized from the reaction of various substituted aldehydes, *β*-naphthol and urea by using Chitosan, a naturally occurring ecofriendly polymer material as catalyst. The structures of all the title compounds were characterized by spectral (IR, ^{1}H, ^{13}C NMR & MASS spectra) and elemental (CHN) analysis. This heterogeneous catalysis had accomplished products with high yields in short reaction times in an eco-friendly approach and the catalyst can also be reused in the synthesis. Hence, this one-pot three component cost-effective protocol has been established as an invariable source for the synthesis of naphtha [1,2-*e*] oxazinones. Chitosan's insolubility, bifunctionality and thermal stability and reusability have elevated its potentiality as heterogeneous catalyst in this protocol.

Keywords: Naphtha-oxazinones, One-pot reaction, Chitosan, *β*-Naphthol, Urea, aldehydes

INTRODUCTION

Heterocycles are the core structural units of majority of pharmaceuticals that are mimicking bio-active natural products. [1] Among them oxazinones and their fused poly nuclear heterocyclic compounds have gained significant consideration of researchers owing to exhibition of an extensive array of medicinal and biological activities [2] like anti-microbial, [3] anti-proliferative, [4] anti-HIV, [5] anti-viral, [6] anti-hypertensive, [7] anti-convulsant, [8] anti-malaria, [9] anti-arrhythmic [10] activities and they also acts as protein kinase inhibitors, [11] platelet fibrinogen receptor antagonists [12] and 5-HT ligand binding inhibitors. [13] Due to these wide range of applications, the chemistry of oxazinones has evoked keen interest in the area of synthetic organic chemistry and there is a essentiality to design and develop new synthetic routes for the synthesis of oxazinone derivatives. Different classes of compounds like salisylaldehyde, [14] amino alcohols, [15] 2-hydroxy-acetophenone, [16] betti bases [17] carbamates, [18] 6-quinolinol [19] and dimedone [20] were used in the synthesis of various types of fused oxazinone derivatives. These compounds have also been employed as precursors to synthesize chiral amino phosphine ligands which are noted asymmetric catalysts to use. [21] Due to these extensive applications, oxazinone chemistry has evoked the interest of organic chemistry researchers.

Based on this importance and need for development of newer oxazinone derivatives, specifically naphtha[1,2-

e]oxazinones have been synthesized from various synthetic routes that involves important catalysts like zinc oxide nanoparticle, [2] perchloric acid supported on silica ($HClO_4/SiO_2$), [22] and silica-bonded *S*-sulfonic acid. [23] However, these methods have their own merits and suffers from some shortcomings like tedious work-up procedures, long reaction times, using of toxic solvents, costly reagents and modest yields. An idea to eradicate these difficulties had prompted us to identify a new and innovative catalyst for the environmentally benevolent synthesis of oxazinones. In such search of potential catalyst, we have succeeded in synthesis of 1,2-dihydro-1-arylnaphtho[1,2-*e*][1,3]oxazine-3-ones from the condensation reaction of *β*-naphthol, urea and aldehydes by using chitosan catalyst.

Chitosan, a natural linear poly-glucosamine composed of randomly distributed *β*-(1→4)-linked D-glucosamine *i.e.*, 2-amino-2-deoxy-D-glucan polysaccharide belongs to a class of hetero polymer. Chitosan's insolubility in organic solvents and water, its bi-functionality (contains 1° & 2° hydroxy and amino groups in higher concentration) elevates its greater potential as heterogeneous catalyst in organic synthesis. Therefore, it is able to activate both electrophilic and nucleophilic accomplices of the reaction by the capability of making hydrogen bonds with electron rich centers and donating its lone pair of electron density to electron deficient centers. The wide spectrum of chitosan's applicability in biology, medicine, and food industries made it as a versatile source. It has been used as a solid catalyst in many of organic name reaction such as Heck reaction, [24] Suzuki cross-coupling, [25] [3+2] Huisgen cyclo-addition, [26] Ullmann reaction, [27] Michael addition reaction, [28] and Aldol & Knoevenagel reactions. [29] Recently chitosan has been used in variety of heterocycles [30] like pyridiazines, [31] fused pyridazines [32] and dienamides. [33] It has also been used as catalyst in asymmetric synthesis. [34,35] This background has created the prominence for chitosan in organocatalysis which does not involve metals and this heterogenization also offers additional advantages like its easy separation from products, reusability, and adoptability for large scale production. This led us to extend the synthesis of naphtha[1,2-*e*]oxazinones from the reaction of araldehydes, phenol and urea by Mannich approach.

MATERIALS & METHODS

A. General:

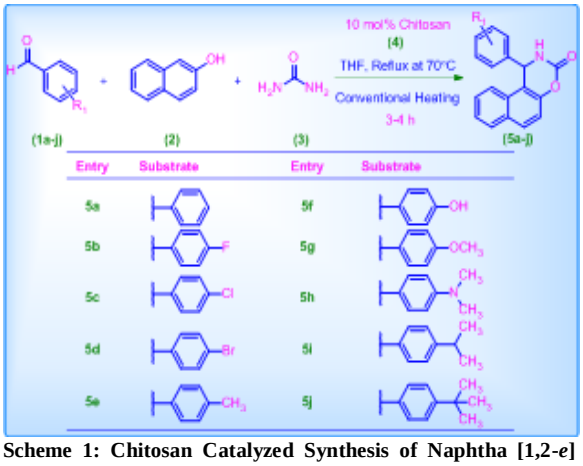
The chemicals were procured from Sigma-Aldrich and solvents were further purified by double distillation processes and used in the reactions. All reactions were performed on Remi magnetic stirrer cum heater. Refluxion was done by setting up reaction flask in oil bath and condensation was done by setting the condenser setup to the reaction flask for water circulation. Melting points of the purified compounds were determined in open capillaries on Guna melting point apparatus and recorded in degree centigrades. The IR spectra were recorded on Bruker FTIR spectrometer equipped with single reflection sampling module and the absorption maxima values were reported in wavenumbers (cm^{-1}). NMR spectra were recorded on Bruker 500MHZ NMR spectrometer by setting operating frequency for ^1H NMR as 500MHZ and for ^{13}C-NMR as 125MHZ by dissolving samples in $CDCl_3$ and referenced to Tetramethylsilane (TMS). Mass spectra were recorded on a JEOL GCMATE II GC-MS spectrophoto-meter at IIT-SAIF, Chennai and C, H, N analysis was done on Thermo Finnigan Instrument.

B. Chitosan catalyzed synthesis of 1,2-dihydro-1-arylnaphtho[1,2-e] [1,3] oxazine-3-ones (5a-j)

The mixture of benzaldehyde (1a, 1 mmol), *β*-naphthol (2, 1 mmol), urea (3, 1.5 mmol) and chitosan (4, 5mol%) was taken in a round bottom flask in THF and refluxed at 70°C for 3h (Scheme 1). The reaction progress was checked with thin layer chromatography (TLC) by using ethyl

acetate and hexane mixture in 1:1 ratio. On completion of reaction, catalyst was collected by filtration and was purified by washing with hot methanol, dried in vacuum oven and reused.

The filtrate containing product was subjected for solvent evaporation under reduced pressure by rota-evaporator and crude 1-phenyl-1*H*-naphtho[1,2-*e*][1,3]oxazin-3(2*H*)-one (5a) was collected and purified by column chromatography by taking ethyl acetate and hexane in 1:2 ratio. The same procedure is adapted for the synthesis of 5b-j and was characterized by their physical, spectral and elemental analysis.



Scheme 1: Chitosan Catalyzed Synthesis of Naphtha [1,2-*e*] oxazinones (5a-j)

C. Spectral & elemental characterization data of 5a-j

1-phenyl-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5a): Yield: 95%; mp: 217-219°C; IR (ZnSe): 3229 (NH), 1748 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 6.11 (1H, s, CH), 7.35-7.92 (11H, m, Ar-H), 8.81 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 52.40, 114.51, 118.63, 124.55, 125.84, 127.03, 127.47, 127.89, 128.33, 128.65, 129.23, 130.09, 131.93, 142.27, 149.04, 152.15; LCMS m/z: 275 (M+); Anal. Calcd for $C_{18}H_{13}NO_2$ (%): C, 78.53; H, 4.76; N, 5.09; Found: C, 78.26; H, 4.70; N, 5.04.

*1-(4-fluorophenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5b):*Yield: 94%; mp: 198-200°C; IR (ZnSe): 3255 (NH), 1754 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 6.25 (1H, s, CH), 7.26-8.13 (10H, m, Ar-H), 8.95 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 53.63, 114.69, 115.24, 119.83, 124.93, 125.84, 126.37, 126.93, 127.45, 130.23, 131.54, 132.26, 137.63, 147.16, 154.93, 161.25; LCMS m/z: 293(M+); Anal. Calcd for $C_{18}H_{12}FNO_2$ (%): C, 73.71; H, 4.12; N, 4.78; Found: C, 73.48; H, 4.04; N, 4.71.

*1-(4-chlorophenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5c):*Yield: 95%; mp: 203-205°C; IR (ZnSe): 3266 (NH), 1736 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 6.26 (1H, s, CH), 7.25-8.19 (10H, m, Ar-H), 8.57 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 53.21, 115.47, 119.03, 125.05, 125.77, 126.33, 127.29, 127.93, 129.21, 129.93, 130.45, 131.64, 132.73, 141.46, 149.59, 154.06; LCMS m/z: 309 (M+); Anal. Calcd for $C_{18}H_{12}ClNO_2$ (%): C, 69.80; H, 3.90; N, 4.52; Found: C, 69.62; H, 3.83; N, 4.46.

1-(4-bromophenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5d): Yield: 93%; mp: 216-218°C; IR (ZnSe): 3227 (NH), 1733 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 6.24 (1H, s, CH), 7.35-8.28 (10H, m, Ar-H), 8.86 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 53.53, 115.44, 119.61, 124.23, 125.85, 126.42, 126.52, 127.24, 129.86, 129.93, 130.65, 131.73, 132.06, 141.23, 149.84, 154.55; LCMS m/z: 353(M+); Anal. Calcd for $C_{18}H_{12}BrNO_2$ (%): C, 61.04; H, 3.41; N, 3.95; Found: C, 60.93; H, 3.33; N, 3.89.

1-(p-tolyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5e): Yield: 95%; mp: 164-166°C; IR (ZnSe): 3262 (NH), 1748 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 2.24 (3H, s, CH_3), 6.13 (1H, s, CH), 7.36-8.05 (10H, m, Ar-H), 8.87 (1H, s, NH), 11.14 (1H, s, OH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 22.35, 54.03, 115.27, 119.94, 124.53, 125.77, 126.49, 127.33, 127.85, 128.48, 130.54, 131.64, 132.24, 137.83, 140.54, 150.21, 154.38; LCMS m/z: 289 (M+); Anal. Calcd for $C_{19}H_{15}NO_2$ (%): C, 78.87; H, 5.23; N, 4.84; Found: C, 78.70; H, 5.14; N, 4.79.

1-(4-hydroxyphenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5f): Yield: 94%; mp: 178-180°C; IR (ZnSe): 3256 (NH), 1752 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 6.26 (1H, s, CH), 7.25-8.06 (10H, m, Ar-H), 8.69 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 53.74, 115.63, 117.25, 120.34, 124.87, 125.83, 126.54, 127.05, 127.46, 129.37, 130.86, 131.95, 133.03, 148.55, 154.70, 155.84; LCMS m/z: 291 (M+); Anal. Calcd for $C_{18}H_{13}NO_3$ (%): C, 74.22; H, 4.50; N, 4.81; Found: C, 74.03; H, 4.42; N, 4.75.

1-(4-methoxyphenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5g): Yield: 92%; mp: 188-190°C; IR (ZnSe): 3235 (NH), 1754 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 3.76 (3H, s, OCH_3), 6.24 (1H, s, CH), 7.24-8.07 (10H, m, Ar-H), 8.73 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 51.77, 57.21, 114.84, 119.33, 124.36, 125.64, 126.43, 126.88, 127.52, 127.94, 129.42, 130.96, 131.63, 132.87, 149.55, 155.15, 156.32; LCMS m/z: 305 (M+); Anal. Calcd for $C_{19}H_{15}NO_3$ (%): C, 74.74; H, 4.95; N, 4.59; Found: C, 74.59; H, 4.89; N, 4.54.

1-(4-(dimethylamino)phenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5h): Yield: 91%; mp: 217-219°C; IR (ZnSe): 3224 (NH), 1734 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 2.94 (6H, s, CH_3), 6.07 (1H, s, CH), 7.13-8.46 (10H, m, Ar-H), 8.79 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 42.06, 53.83, 113.55, 116.15, 117.27, 123.63, 125.43, 126.45, 127.53, 128.08, 129.24, 130.23, 131.54, 132.86, 146.68, 149.65, 155.05; LCMS m/z: 318(M+). Anal. Calcd for $C_{20}H_{18}N_2O_2$ (%): C, 75.45; H, 5.70; N, 8.80; Found: C, 75.28; H, 5.62; N, 8.73.

1-(4-iso-propylphenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5i): Yield: 90%; mp: 179-181°C; IR (ZnSe): 3186 (NH), 1745 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 1.64 (6H, s, $(CH_3)_2$), 2.85 (1H, m, CH), 6.17 (1H, s, CH), 7.23-8.09 (10H, m, Ar-H), 8.83 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 22.46, 33.55, 52.83, 115.85, 118.74, 124.47, 125.53, 125.86, 126.08, 127.24, 127.86, 130.05, 131.93, 132.44, 141.96, 148.27, 150.35, 154.87; LCMS m/z: 317 (M+); Anal. Calcd for $C_{21}H_{19}NO_2$ (%): C, 79.47; H, 6.03; N, 4.41; Found: C, 79.28; H, 5.95; N, 4.36.

1-(4-(tert-butyl)phenyl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one (5j): Yield: 91%; mp: 178-180°C; IR (ZnSe): 3257 (NH), 1732 (C=O) cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$, ppm) δ: 1.25 (6H, s, CH_3), 1.37 (3H, s, CH_3), 6.34 (1H, s, CH), 7.43-8.26 (10H, m, Ar-H), 8.96 (1H, s, NH); ^{13}C-NMR (125 MHz, $CDCl_3$, ppm) δ: 32.07, 35.63, 53.44, 114.88, 118.47, 124.23, 125.84, 125.98, 126.87, 127.27, 128.46, 131.54, 132.45, 132.74, 140.25, 150.25, 151.06, 154.67; LCMS m/z: 331 (M+). Anal. Calcd for $C_{22}H_{21}NO_2$ (%): C, 79.73; H, 6.39; N, 4.23; Found: C, 79.55; H, 6.32; N, 4.18.

RESULTS & DISCUSSION

A. Catalyst optimization for the synthesis of 5a-j

Synthesis of 1-phenyl-1*H*-naphtho[1,2-*e*][1,3]oxazin-3(2*H*)-one (5a) as model reaction to identify the effective catalyst for its formation in high yields with short reaction times, we have investigated the effectiveness of various types of catalysts like Lewis acids, Bronsted acids, solid acids, clay catalysts, solid supported catalysts, metal catalysts, metal complexes and nano materials *viz.*, Cyanuric chloride [2,4,6-Trichloro-1,3,5-triazine], PTSA [*p*-Toluenesulfonic acid], Molecular Iodine, Montmorillonite K-10, Dichlorotris (triphenyl phosphine) ruthenium(II) $[RuCl_2(PPh_3)_3]$ complex, $Yb(OTf)_3$ [Ytterbium(III) trifluoromethanesulfonate/ ytterbium(III) triflate], TEBA [Triethyl-benzylammonium chloride], $FeCl_3$-SiO_2-NP [silica supported ferric chloride nanoparticles], $TiCl_4$/Sm [Samarium supported Titanium tetrachloride], ZnO-NP [Zincoxide nanoparticles], $HClO_4/SiO_2$ [Silica Supported Perchloric Acid] & chitosan [poly-(*D*)glucosamine, a hetero polymer] as enlisted in Table 1. All the reactions were performed in THF solvent at

same operating temperatures with 10mol% of enlisted catalyst concentrations to accomplish 5a in good yields and were provided in Table 1 against the corresponding investigated catalysts. The obtained yields of 5a with chitosan catalyst are comparatively good (95%) than other catalysts that are investigated. This is due to the physical and chemical characteristics of chitosan have elevated the reactivity of the substrates to form products. Hence we have considered chitosan as a cost-effective and eco-friendly catalyst that involved with simple reaction procedures and hence further we proceeded for other parameters like catalyst concentration optimization, reaction temperature optimization and catalyst reusability.

Table 1: Optimization of catalyst

S.No.	Catalyst	Time (h)	Yield (%)
1	Cyanuric Chloride	5	87
2	*p*-TSA	4	89
3	Molecular Iodine	5	82
4	Montmorillonite K-10	7	80
5	$Yb(OTf)_3$	6	84
6	$RuCl_2(PPh_3)_3$	3.5	92
7	TEBA	6	86
8	$TiCl_4$/Sm	5	85
9	ZnO-NP	4.5	87
10	SiO_2-$FeCl_3$-NP	4	90
11	$HClO_4$ /SiO_2	5	83
12	Chitosan	3	95

B. Optimization of catalyst concentration

Synthesis of 5a has been accomplished in 85, 90, 95, 95, 95, 94% yields from reactions that were loaded with 1.0, 2.5, 5.0, 7.5, 10.0 and 12.5 mol% of chitosan catalyst (4) respectively which were operated at same reaction conditions. It is observed that 5mol% of chitosan is effective one in accomplishing 5a in 95% yield and higher concentrations have not shown any positive effect (Table 2) on improving the yields.

Table 2: Optimization of catalyst concentration

S.No.	Catalyst (mol%)	Time (h)	Yield (%)
1	1.0	6	85
2	2.5	5	90
3	5.0	3	95
4	7.5	4	95
5	10.0	4	95
6	12.5	4	94

The higher concentrations of catalyst (7.5, 10.0 & 12.5 mol%) were identified as unproductive compared to 5.0 mol% due to the fact that the catalyst molecules will spread over solvent and hence disturbs the interaction of substrate molecules and ultimately retards the reactivity of substrates. It can be clearly observed in the case of 7.5 & 10.0 mol% of catalyst involving reactions as they have shown the same effect as 5.0 mol% and 12.5 mol% of catalyst retards more the interaction of substrates and the yield has been decreased. Hence the higher catalyst concentrations have not shown any effect on enhancement of the product yields.

C. Reusability of the catalyst

Reusability of the chitosan for the synthesis of 5a has been investigated with the above optimized conditions. After use and filtration of chitosan in the reaction, it was purified by washing with hot ethanol for 2-3 times and dried over a period of 8-10h in vacuum oven and reused for six successive runs (Figure 1) and 5a has been obtained in 95, 95, 93, 89, 84, 78 & 71% of yields and effective reusability of the catalyst has been considered up to three runs.

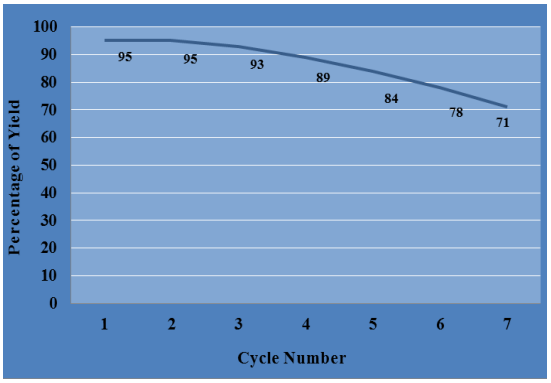


Figure 1: Reusability of the catalyst

D. Catalytic activity of Chitosan

Due to the presence of free -NH_2 & -OH groups in chitosan (Figure 2), it acts as a mild bifunctional heterogeneous catalyst. Its insolubility in most of the solvents elevates its greater potential as heterogeneous catalyst and activates nucleophilic and electrophilic centers of the reaction substrates to fulfill the

requirements for this multicomponent synthesis of naphtha[1,2-*e*] oxazinones.

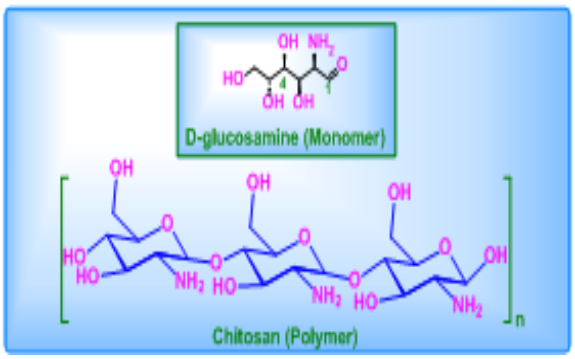


Figure 2: Structure of Chitosan and its active catalytic sites

E. Mechanistic aspects of the Chitosan catalyzed synthesis of Naphtha [1,2-e] oxazinones

The -NH_2 hydrogens of the chitosan form the intermolecular hydrogen bonding with the carbonyl oxygen of the aldehyde and enhance the electrophilicity of carbonyl carbon and get facilitated to react with urea. Then the formed 1-(hydroxyl (aryl) methyl) urea will undergo dehydration and forms an imino compound *viz.*, 1-(aryl-1-yl methylene)urea. In the same way chitosan by donating the lone pair electron density of -NH_2 group to electophilic carbon and polarizes the imino bond (C=N) of 1-(aryl-1-yl methylene)urea and enhances the comparative electropositivity on imino carbon. This facilitates the nuclophilic attack of *β*-naphthol (in its oxonium resonated form) on iminium carbon and made them to react and then the intramolecular proton transfer forms the 1-((2-oxo-1,2-dihydronaphthalen-1-yl)(phenyl)methyl)urea, it on expulsion of ammonia forms the desired 1-Aryl-1,2,4a,10b-tetrahydro-3*H*-naphtho[1,2-*e*][1,3] oxazin-3-ones (Figure 3). In this way the chitosan catalyst has been bifunctionally involved in the reaction and promoted the synthesis of 1,2-dihydro-1-arylnaphtho[1,2-e][1,3]oxazine-3-ones.

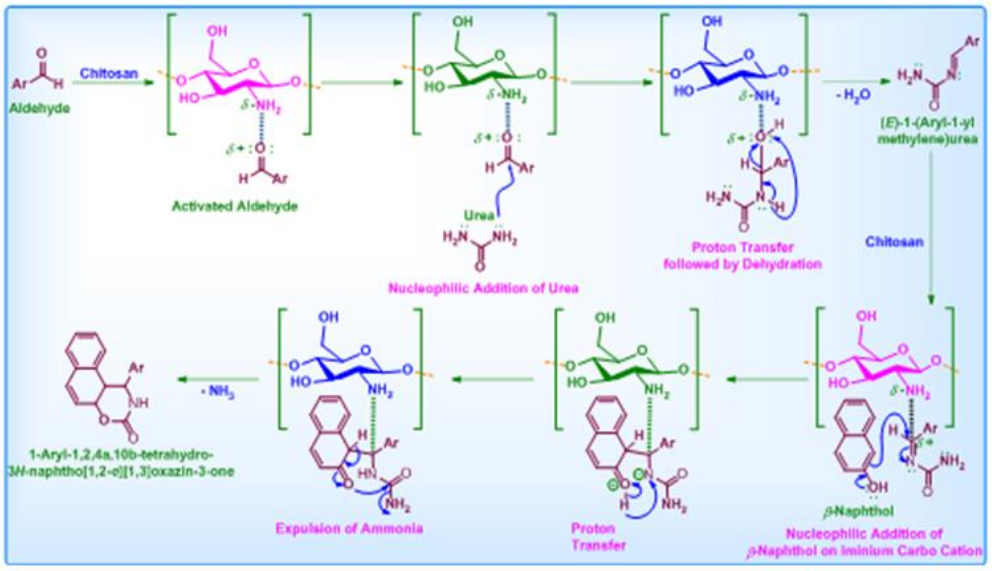


Figure 3: Plausible mechanism for the synthesis of Naphtha [1,2-e] oxazinones (5a-j)

CONCLUSION

In summary we have established an efficient and environmentally benign methodology for the synthesis of a series of 1,2-dihydro-1-aryl-naphtho[1,2-*e*][1,3]oxazine-3-ones from the one-pot three-component reaction of various substituted aldehydes, *β*-naphthol and urea in the presence of chitosan under conventional heating by using chitosan catalyst. The advantages of this ecofriendly method are reduced reaction times, experimental simplicity, simple work-up for purification, higher yields and reusability of the catalyst.

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