

Review Article

Aminonaphthoquinone: A Versatile Synthron for the Synthesis of Naphthoquinone-fused *N*-heterocycles via Multicomponent Reactions (MCRs)

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Abstract

The naphthoquinone-fused *N*-heterocycles are found in natural products and are important candidates in medicinal chemistry. The 1, 3-C, N-binucleophilic property of the 2-aminonaphthoquinone provides access for the synthesis of naphthoquinone-fused *N*-heterocycles. In recent years this property of 2-aminonaphthoquinone has been acknowledged greatly by the synthetic community for construction of variably substituted naphthoquinone-fused *N*-heterocycles. This review summarizes the important findings in this area during 2019-2024. Also the review highlights the gap and future perspective of the approach.

Keywords

Aminonaphthoquinone, Fused Pyrrole, Fused Dihydropyridine, Multicomponent Reactions, 1, 3-C, N Binucleophile

1. Introduction

Heterocycles is a significant class of organic molecules with diverse applications [1-11]. Among the heterocycles the *N*-heterocycles [12-14], are celebrated owing to their dominance in natural products, medicinal chemistry and drug discovery [15-18]. About 75% of the FDA-approved drugs contain azaarenes core [19, 20]. The *N*-heterocycles show a broad spectrum of bioactivities such as anticancer, antimalarial, anti-HIV, anti-tubercular, anti-diabetic, antiviral, anti-inflammatory, anti-convulsant activities [19-24]. Apart from the medicinal dominance *N*-heterocycles also find application as chemosensor for metal cation and anion detection [25, 26], solar cell [27]. Thus discovering green and sustain-

able approaches for the synthesis of functionalized *N*-heterocycles is always a challenge to the synthetic organic chemists. Figure 1 displays some representative examples of *N*-heterocycles that are present in natural product and show several bioactivities [16, 17, 19, 20]. The Hyacinthacine A₁ (1) and (-)-epiquinamide (2) are natural products. The ezetimibe (3) and clavulanic acid (4) those contain β -lactam moiety show cholesterol absorption inhibitory and which β -lactamase inhibitory activities respectively. The triazole containing carboxyamidotriazole (5) is an anticancer agent. The dacarbazine (6) which contains an imidazole ring shows antifungal as well as anticancer activity.

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Received: 25 May 2025; Accepted: 11 June 2025; Published: 28 August 2025



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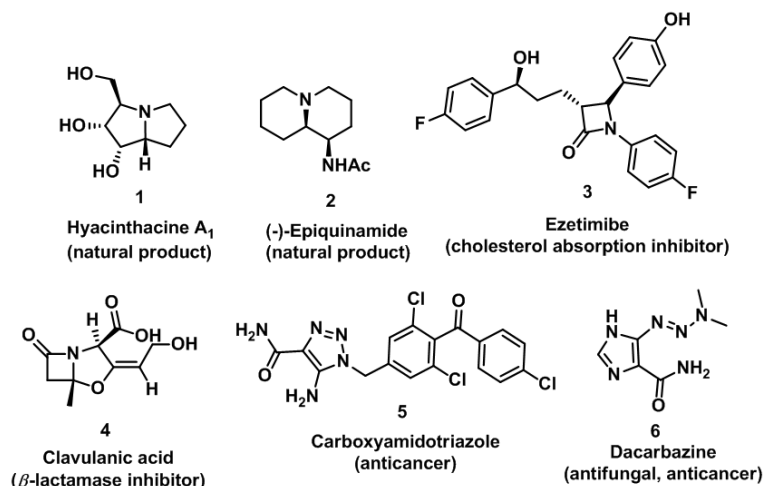
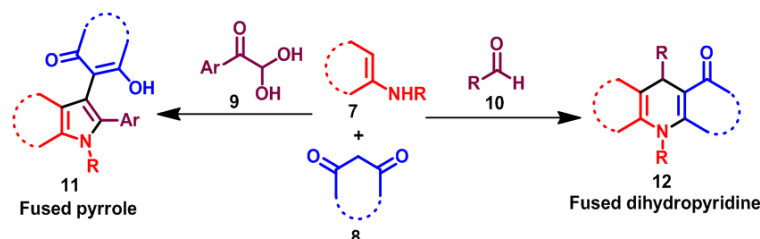


Figure 1. Representative examples of some naturally occurring and bioactive N-heterocycles.



Scheme 1. General reaction scheme for synthesis of fused pyrrole and fused dihydropyridine from aminoarene.

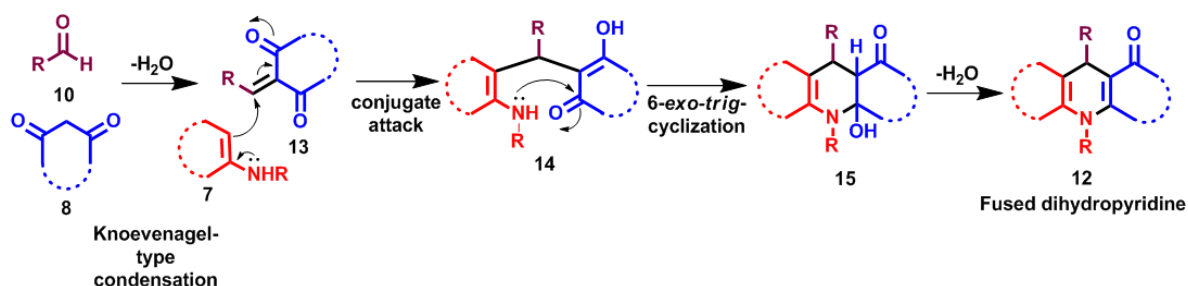
The increasing pollution from the traditional organic synthesis demands the discovery of alternative synthesis by minimizing the waste generation, employing green solvents, catalysts, safe-handling substrates and green energy sources. All these aspects combiningly called green chemistry the term coined by P. Anastas and R. Garret in 1991 [28-33]. Several alternative synthetic approaches such as microwave mediated synthesis, visible light-assisted synthesis, ultrasound-assisted synthesis, grindstone chemistry, ball milling and electrochemical approaches have gained popularity over the years [34-36].

The multicomponent reactions (MCRs) [37-43] have gained popularity in this context. The MCRs are one-pot reactions that involve the reactions of at least three substrates in a stereo and regioselective manner to produce the products that contains maximum contributions from the reactants.

MCRs have the ability to provide molecular complexity in one-pot. Thus MCRs are one of the preferred routes over multi step synthesis.

Thus the synthesis of heterocycles via MCRs has remained a hot topic in synthetic research. In this context, the aminoarenes are important substrates that possess 1, 3-C, N-binnucleophilic property which provide access for fused pyrroles [44-47] and dihydropyridines [48-50] depending on the use of arylglyoxal and aldehyde respectively (Scheme 1).

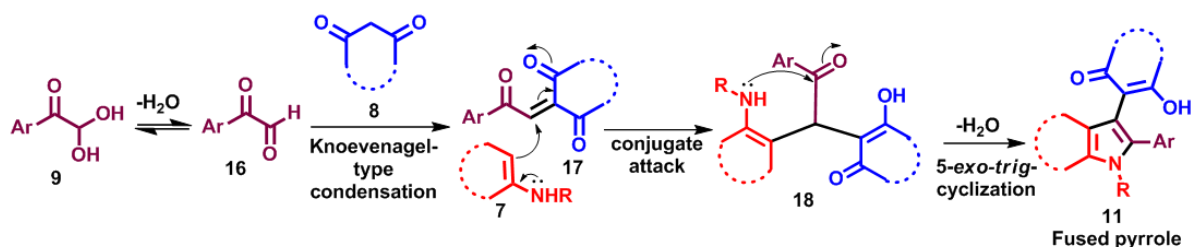
The common pathway followed for the synthesis of fused dihydropyridines is shown in the Scheme 2. The aldehyde and the 1, 3-dicarbonyl undergoes Knoevenagel-type condensation to form the alkene intermediate. The alkene intermediate is then attacked by the aminoarene to form the adduct. Intramolecular cyclization followed by dehydration results in the formation of fused dihydropyridine (Scheme 2).



Scheme 2. General reaction pathway for the synthesis of fused dihydropyridine.

In case of fused pyrroles, at first the condensation of aryl glyoxal and the 1, 3-dicarbonyl in a Knoevenagel fashion generates the alkene intermediate. The Michael attack of the

aminoarene generates the adduct. Then the condensation between the carbonyl group of aryl glyoxal and amine group of aminoarene gives the fused pyrroles (Scheme 3).



Scheme 3. General reaction pathway for the synthesis of fused pyrrole.

The 1, 4-naphthoquinone (1, 4-NQ) [51-54] is a privileged molecule which is present in the core of many natural products and is popular in drug discovery. Likewise, the naphthoquinone-fused *N*-heterocycles are important class of molecules present in natural products and shows diverse bioactivities such as anticancer, antifungal, antiviral activities [55]. Figure 2 represents some useful naphthoquinone fused *N*-heterocycles with natural occurrence and bioactivities. Utahmycin B (I), [56] bhimacin D (II) [55], [57] contain naphthoquinone fused pyrroles are naturally occurring molecules. Likewise, compound III, is an anticancer agent which displayed significant ED_{50} values against several human cancer cell lines such as ED_{50} : 0.61 $\mu\text{g/ml}$ against A549

(human lung), ED_{50} : 0.78 $\mu\text{g/ml}$ against SK-OV-3 (human ovarian), ED_{50} : 0.71 $\mu\text{g/ml}$ against HCT 15 (human colon), and ED_{50} : 0.75 $\mu\text{g/ml}$ XF 498 (human CNS) [58]. Similarly, compound IV displays cytotoxic activity against HeLa cells and KB cells with IC_{50} values 0.50, and 0.68 $\mu\text{g/ml}$ respectively [59]. The naphthoquinone-fused dihydropyridine V exhibits promising anticancer potential against human cancer cell lines, IC_{50} : 0.60 ± 0.01 μM against human carcinoma (KB), IC_{50} : 1.05 ± 0.02 μM against human hepatocellular carcinoma (HepG2), IC_{50} : >5.00 μM against lung cancer (A549), IC_{50} : 2.12 ± 0.04 μM against breast carcinoma (MCF7), and IC_{50} : 3.68 ± 0.02 μM against human embryonic kidney (Hek-293) [59].

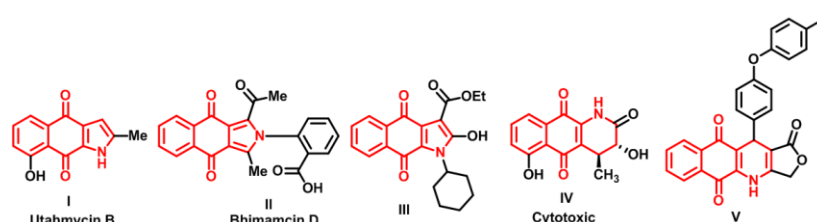


Figure 2. Some naturally occurring and bioactive naphthoquinone fused *N*-heterocycles.

Thus, synthesizing naphthoquinone-fused *N*-heterocycles is an important challenge to the synthetic community. Thus substantial attention was devoted in synthesizing the naphthoquinone fused heterocycles in recent years. In this context, the 2-aminonaphthoquinone is an interesting molecule having two nucleophilic sites (Figure 3) and is a molecule of choice for the synthesis of naphthoquinone fused *N*-heterocycles.

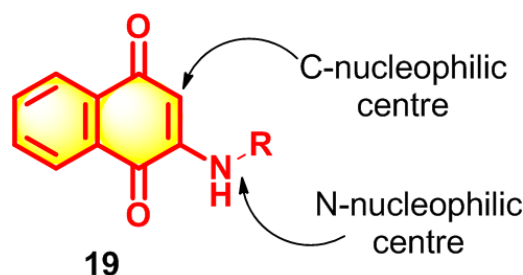


Figure 3. C- and N- nucleophilic sites of 2-aminonaphthoquinone.

Since 2019, aminonaphthoquinone has received a great

deal of attention for synthesizing naphthoquinone-fused *N*-heterocycles by utilizing its 1, 3-C, N-binnucleophilic property. Several research groups have greatly contributed to this field. The aminonaphthoquinone molecule possesses great potential. Thus, we present this review describing the use of aminonaphthoquinone in MCRs for the synthesis of naphthoquinone-fused *N*-heterocycles (Figure 4). This review addresses the state of art and future perspective of aminonaphthoquinone.

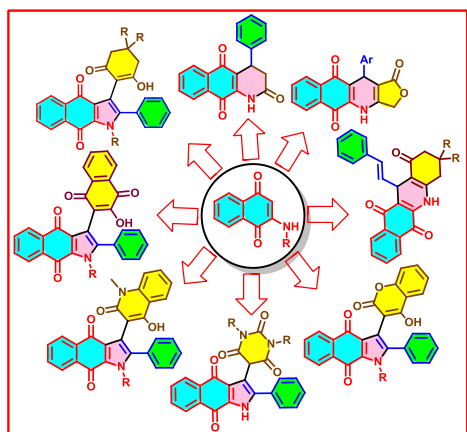
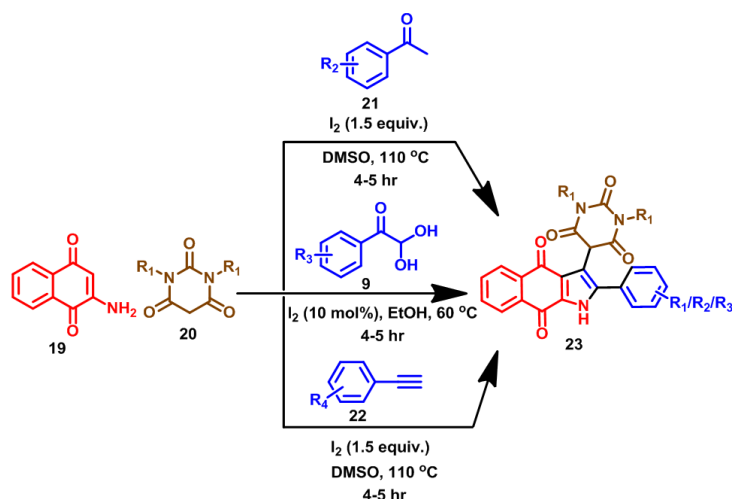


Figure 4. Application of 2-aminonaphthoquinone for the synthesis of naphthoquinone fused *N*-heterocycles.

2. Naphthoquinone-fused Five Membered *N*-heterocycles

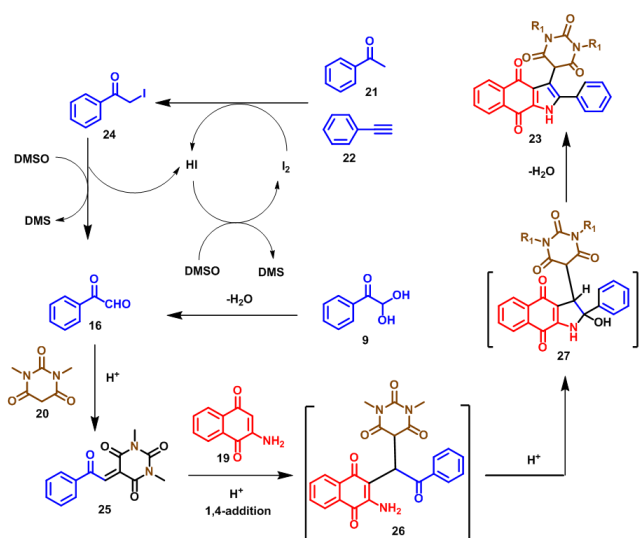
In 2020, Pandey and Choudhury *et al.* reported three approaches for the synthesis of barbituric acid linked naphthoquinone-fused pyrroles **23** (Scheme 4) [55]. The one-pot reaction of 2-aminonaphthoquinone **19**, 1, 3-dimethyl barbituric acid **20** and acetophenones or phenylacetylenes **22** resulted the naphthoquinone-fused pyrroles **23** in presence of

1.5 equivalent I_2 in DMSO medium at 110 °C. In the optimization study several iodine sources such as NaI, KI, TBAI, and I_2 were screened but the I_2 was found as the best iodine source. Also, the stoichiometry of iodine was found critical for the reaction. Increasing the amount of iodine from 1.0 equiv. to 1.5 equiv. the yield of the product increased from 60% to 79%, and the reaction time decreased from 10 hours to 4.5 hours. Further, decreasing the amount of iodine to 0.5 equiv. the lower yield 35% of the product was isolated in 10 hours. The reaction did not proceed in DMF, methanol, ethanol, acetonitrile, dioxane, and toluene. DMSO is necessary for the reaction. The acetophenone with electron donating and electron-withdrawing substituents reacted smoothly in the condition affording good yield of the desired products. Also, when the acetophenone is replaced with the phenylacetylenes with electron-donating as well as electron-withdrawing groups the reaction proceeded smoothly affording good yield of the product. An alternative pathway for the synthesis of the product was also reported which involves the one-pot three component reaction of arylglyoxals **9**, 2-aminonaphthoquinone **19** and barbituric acid **20** to afford the desired product **23**. As per the second optimization study, the catalyst-free approaches were not fruitful in forming the desired product. Employment of iodine (5 mol%) in methanol at 65 °C, afforded the desired product in 48% yield in 6 hours. Increasing the iodine loading to 10 mol%, the product was isolated in 80% in 4 hours. Interestingly, on further increasing the iodine amount to 20 mol%, the product yield decreased to 60%. Thus 10 mol% of iodine was found as the optimum amount. Next, screening of other solvents such as acetonitrile, DMF, and DMSO apart from methanol did not offer better results. With this optimized condition, the scope of arylglyoxals with different stereoelectronic nature was found satisfactory. Also, the scope of the unsubstituted barbituric acid was studied which provided 82% yield of the corresponding product.



Scheme 4. Panday's approach for the synthesis of naphthoquinone-fused pyrroles.

As per the proposed reaction pathway (Scheme 5), Panday and Choudhury reported that the phenyl acetylene or the acetophenone in presence of I_2 -DMSO medium oxidises to form the aldehyde 16 via the iodinated intermediate 24. The aldehyde 16 is also formed by the aryl glyoxal 9 by dehydration. The aldehyde 16 now condensed with the barbituric acid 20 leading to the formation of the alkene intermediate 25. To the intermediate 25, the aminonaphthoquinone 19 attacked in a conjugate manner to form the intermediate 26. Intramolecular cyclization and dehydration finally lead to the end product 23.

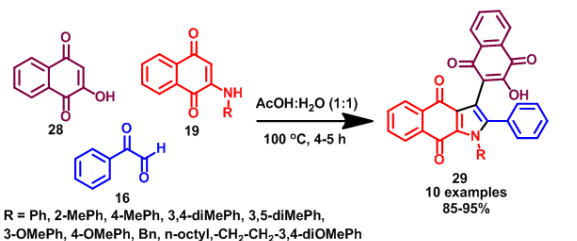


Scheme 5. Reported mechanism for the synthesis of naphthoquinone-fused pyrroles.

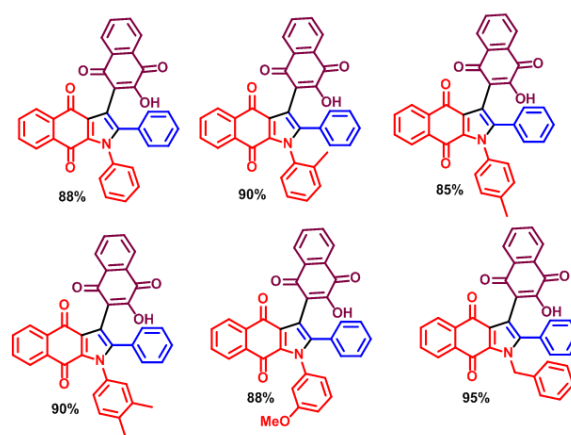
In 2023 Alipoor *et al.* reported a multicomponent synthetic approach for lawsone-linked naphthoquinone-fused pyrroles (Scheme 6) [60]. The three component reaction of lawsone 28, aminonaphthoquinones 19 and the aldehyde 16 in acetic acid water mixture (1: 1) at 100 °C result the naphthoquinone-fused pyrroles 29 in excellent yields (85-95%) in 4-5 hours. The reaction system tolerated a widely alkyl and aryl *N*-substituted aminonaphthoquinone derivatives.

The 1, 1, 1, 3, 3, 3-hexafluoroisopropanol (HFIP) [61-67] has gained popularity in contemporary organic synthesis owing to its unique properties such as H-bond donation, highlyacidic nature, stabilizing ionic species etc. In C-H activation it is greatly acknowledged as a versatile solvent. Chaurasia and Parvin in the same year reported the one-pot three component reactions of 2-aminonaphthoquinones 19, 4-hydroxycoumarins 30 and aryl glyoxals 9 in HFIP medium at 60 °C for the synthesis of naphthoquinone-fused pyrroles in good yields (Scheme 7) [68]. Both the primary and secondary aminonaphthoquinones were reacted well in the reaction medium. During the optimization study, several solvents such as EtOH, MeOH, H_2O , DCE, MeCN, toluene, THF, and HFIP were screened under catalyst-free conditions. Among all the screened solvents HFIP gave best results in terms of yield in

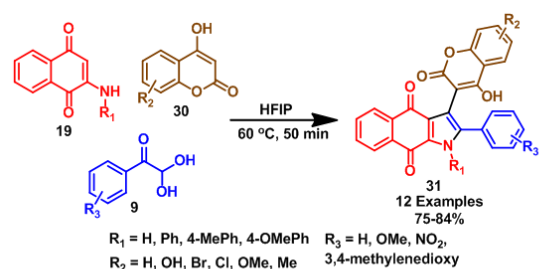
short reaction time. Various electron donating and withdrawing substituents on the phenylglyoxal were found suitable for the reaction. Also the substituents on the coumarin ring were well tolerated.



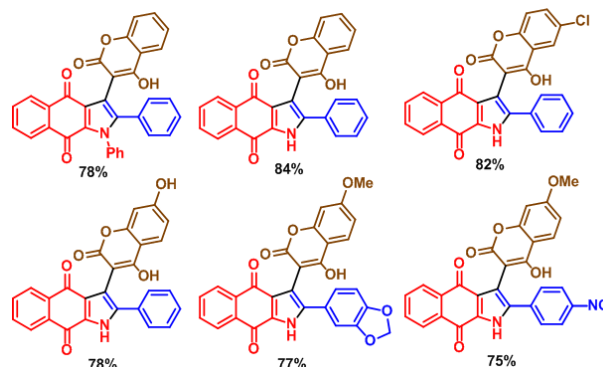
Representative examples



Scheme 6. Reported mechanism for the synthesis of naphthoquinone-fused pyrroles.

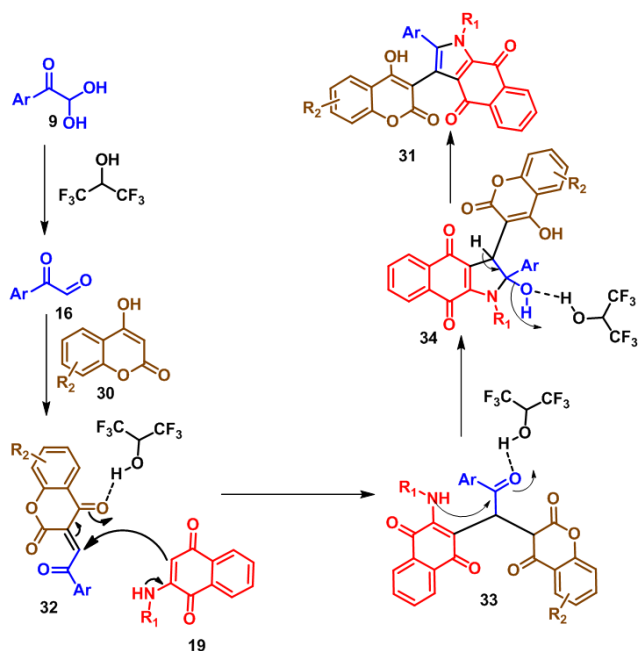


Representative Examples



Scheme 7. HFIP-mediated synthesis of naphthoquinone-fused pyrroles.

As per the report, the reaction proceeds via the pathway as shown in the Scheme 8. At first the HFIP activates the carbonyl group of the aryl glyoxal. Intermediate 32 was formed by the reaction with the 4-hydroxycoumarin 30, which was activated by the HFIP. The conjugate attack of the aminonaphthoquinone 19 formed the intermediate 33. Cyclization and dehydration resulted in the formation of the final product 31.

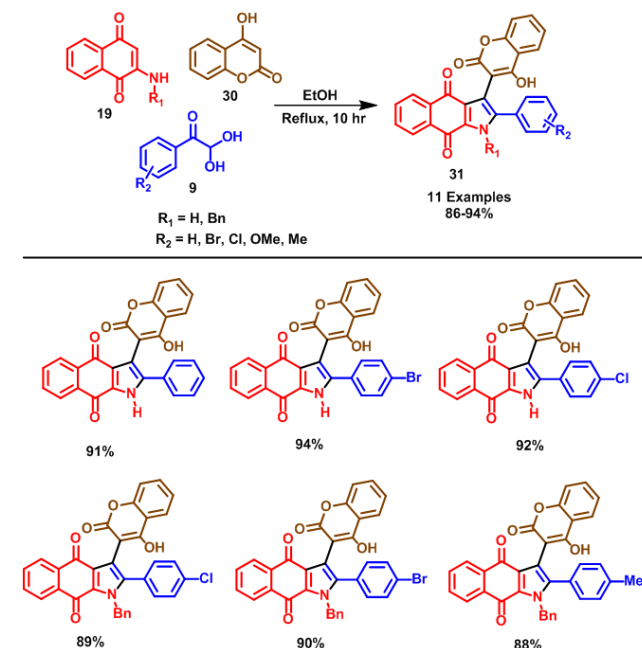


Scheme 8. Reported mechanism for the HFIP mediated synthesis of naphthoquinone-fused pyrrole.

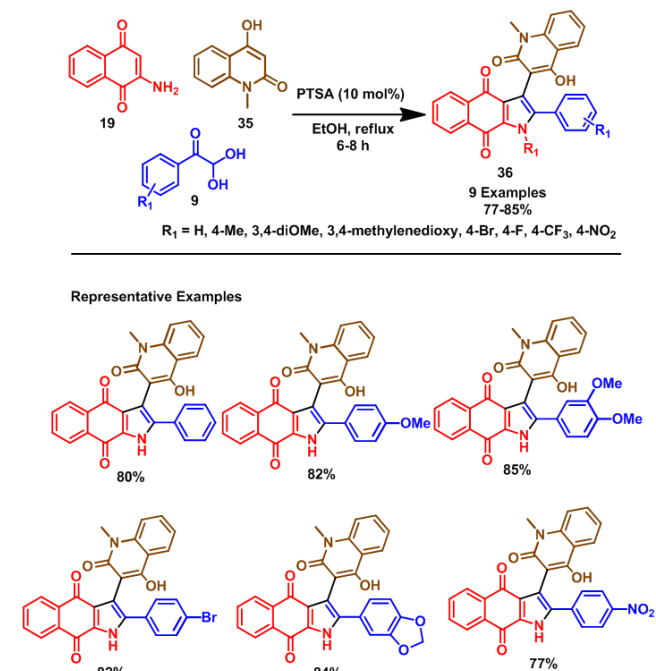
In the same year Alizadeh and co-workers reported another approach for the synthesis of the same scaffold. One-pot reaction of 2-aminonaphthoquinone 19, 4-hydroxycoumarin 30 and arylglyoxal 9 derivatives in ethanol medium under reflux condition afforded the naphthoquinone-fused pyrroles 31 in 86-94% yield (Scheme 9) [69]. Substituted phenyl glyoxals with electron-donating as well as the halogen substituents reacted smoothly to afford the desired product.

In 2023, Panday *et al.* reacted 2-aminonaphthoquinone 19, 4-hydroxy-1-methylquinolin-2(1H)-one 35 and aryl glyoxals 9 in presence of 10 mol% PTSA as catalyst in EtOH medium under reflux condition to synthesize the naphthoquinone-fused pyrroles 36. The products were formed in good to excellent yields within 6-8 hours (Scheme 10) [70]. Irrespective of the stereoelectronic nature of the substituents on the phenyl ring of aryl glyoxal the products were isolated in good

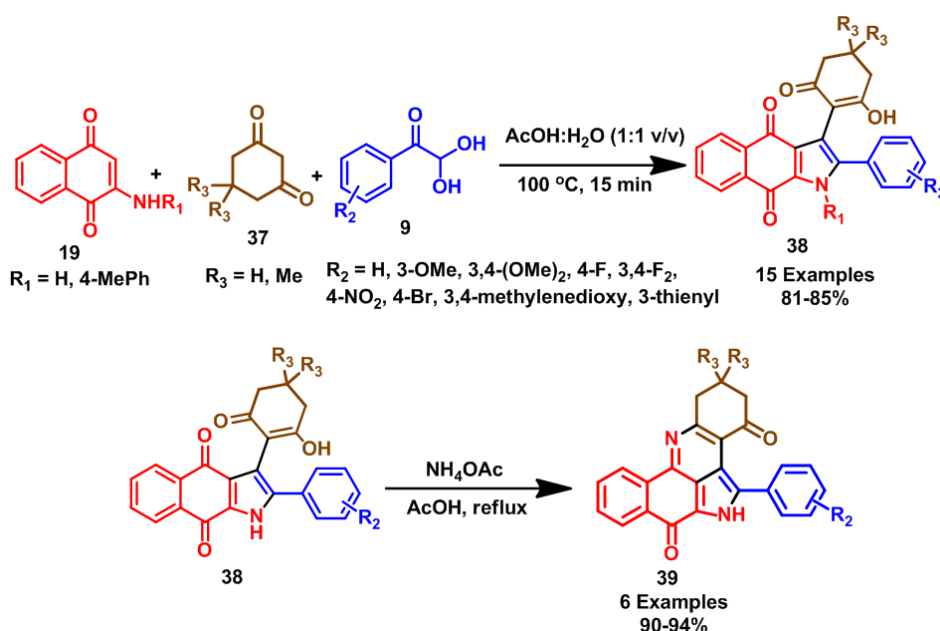
yields.



Scheme 9. Ethanol mediated synthesis of naphthoquinone-fused pyrroles.



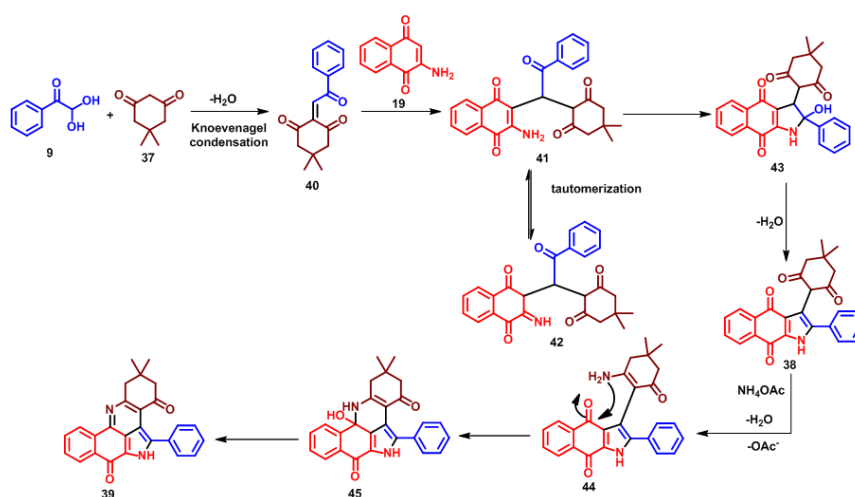
Scheme 10. PTSA catalysed synthesis of naphthoquinone-fused pyrroles.



Scheme 11. AcOH: H₂O mediated synthesis of naphthoquinone fused pyrroles.

Scheme 11 depicts the reaction of 2-aminonaphthoquinone 19, dimedone/cyclohexane-1,3-dione 37, and arylglyoxals 9 in AcOH:H₂O which provided the desired products in very good yields in short reaction time. The electron-donating as well as electron-withdrawing groups on phenylglyoxal, bulky, and heteroaryl glyoxals were also found reactive. Interestingly, when the synthesized products were reacted with NH₄OAc in acetic acid under reflux condition, the complex molecule 39 was isolated in excellent yields (Scheme 11) [71]. As per the

proposed reaction pathway by Chaurasia and Parvin, at first the dimedone and arylglyoxal undergoes Knoevenagel condensation to form the intermediate 40. Then the nucleophilic attack of aminonaphthoquinone 19 generates the intermediate 41. Next, intermediate 41 undergoes intramolecular cyclization and dehydration to form the product 38. The reaction of NH₄OAc with 38 generates the intermediate 44, which again on intramolecular cyclization and dehydration forms the product 39 (Scheme 12).



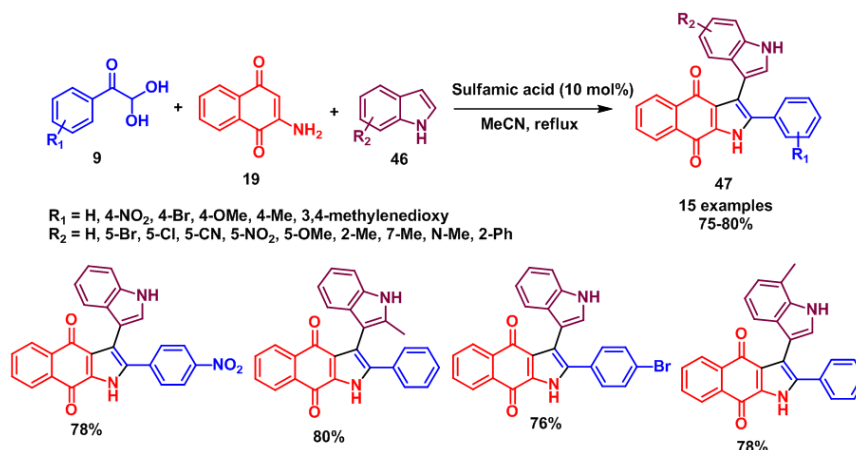
Scheme 12. Proposed reaction mechanism.

Indole, owing to its promising bioactivities [72] and presence in numerous natural products [73] is a popular moiety for medicinal chemists and biologists. Also, its C-3 nucleophilicity has been utilized in multicomponent reactions for constructing biologically active complex molecular scaffolds

[74, 75]. In this context, Parvin group have explored the nucleophilicity of indole to construct indole-linked naphthoquinone-fused pyrroles 47 from the one-pot three component reactions of arylglyoxals 9, 2-aminonaphthoquinone 19 and substituted indoles 46 in acetonitrile in the presence of sul-

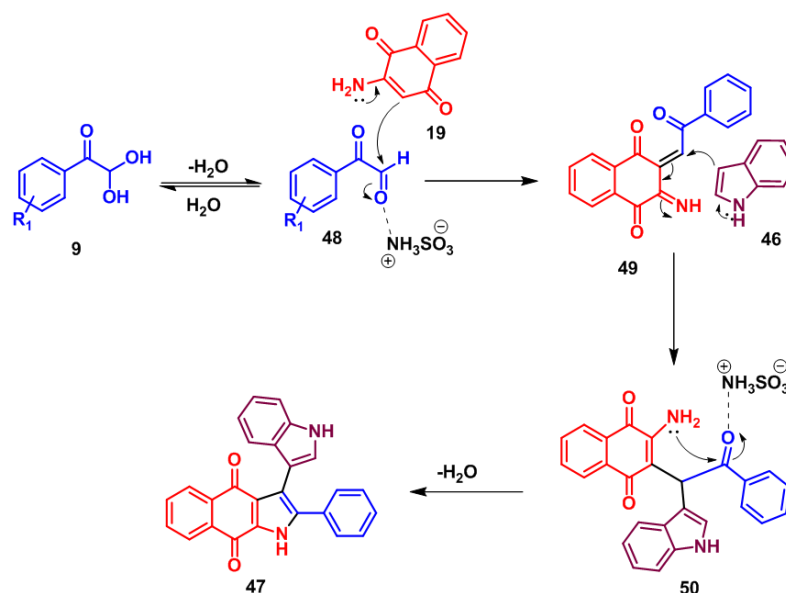
famic acid (10 mol%) which afforded the desired products in 75-80% (Scheme 13) [76]. Several electron-donating and electron-withdrawing substituents on indole such as 5-Br, 5-Cl, 5-CN, 5-NO₂, 5-OMe, 2-Me, 7-Me, N-Me, 2-Ph were found suitable in the reaction conditions providing very good

yields. Likewise, the electron-donating as well as electron-withdrawing substituents on the phenylglyoxal were also proceeded smoothly to afford the desired products. The protocol was also applied for the synthesis of the product in gram-scale.



As displayed in Scheme 14, the arylglyoxal in the presence of sulfamic acid the activated aldehyde 48 was attacked by 2-aminonaphthoquinone 19 to generate the Michael acceptor

49. Then, the Michael addition of indole 46 formed the adduct 50 which upon 5-*exo-trig*-cyclization and dehydration formed the desired product 47.

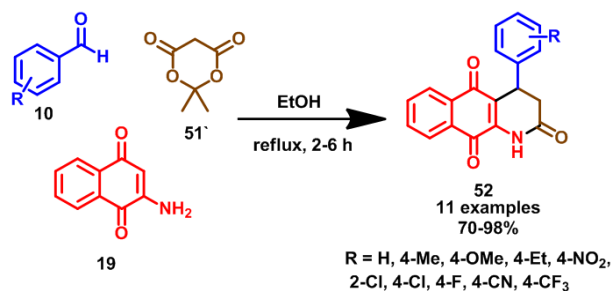


3. Naphthoquinone-fused six Membered N-heterocycles

The meldrum's acid is an important candidate in MCRs. Meldrum's acid provides access to the diverse functionalized

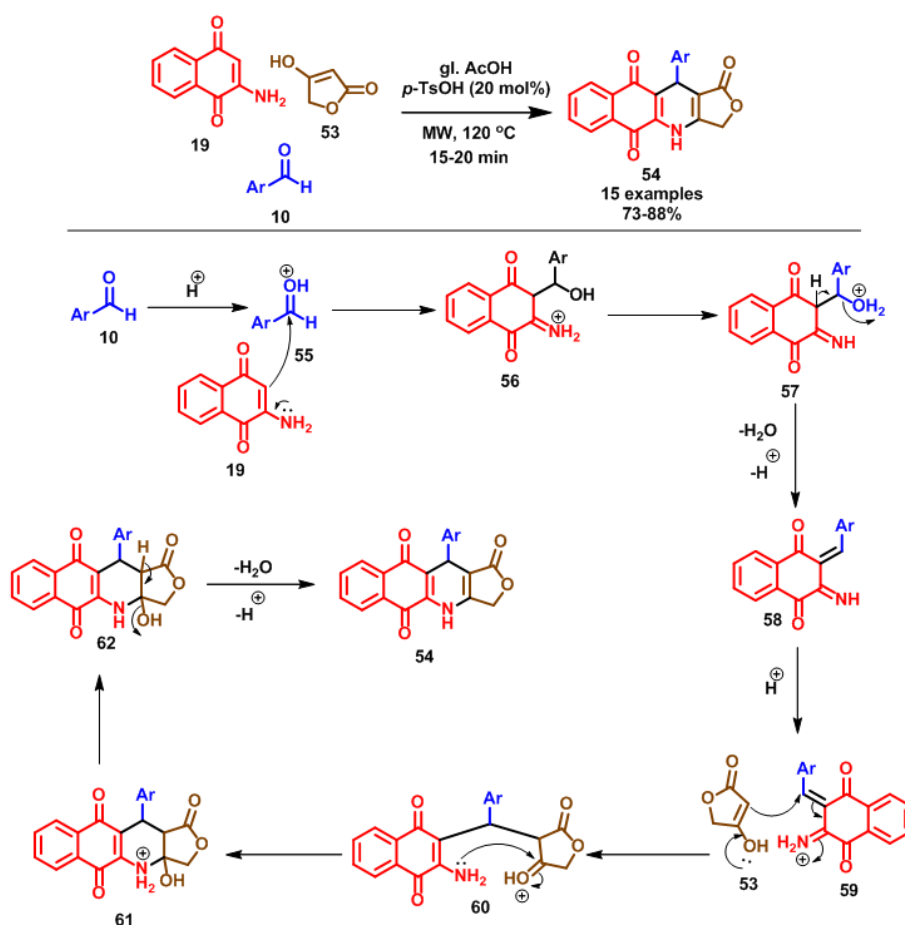
fused heterocycles. Thus, its use in MCRs has increased in the last decade [77]. The catalyst-free ethanol mediated synthesis of 4-aryl-3, 4-dihydrobenzo [g] quinolone-2, 5, 10(1H)-triones 52 under reflux condition has been reported by Kamalifar and Kiyani *et al.* in 2019 (Scheme 15) [78]. The one-pot three component reaction of 2-aminonaphthoquinone 19, Meldrum's acid 51 and aromatic aldehydes 10 in EtOH

under reflux result the final products 52. Asymmetric synthesis [79-83] is an important strategy to synthesize the chiral molecules with high enantiomeric excess, which often finds applications in medicinal chemistry and drug discovery. In recent years, there are numerous asymmetric versions of multicomponent reactions have been developed employing asymmetric metal, and organocatalysts [84-87]. Since the compound 52 contains a chiral centre, employing asymmetric catalysts may provide the product with good enantiomeric excess and which may show potential bioactivities in future.



Scheme 15. Catalyst-free synthesis of 4-aryl-3, 4-dihydrobenzo [g] quinolone-2, 5, 10(1H)-triones.

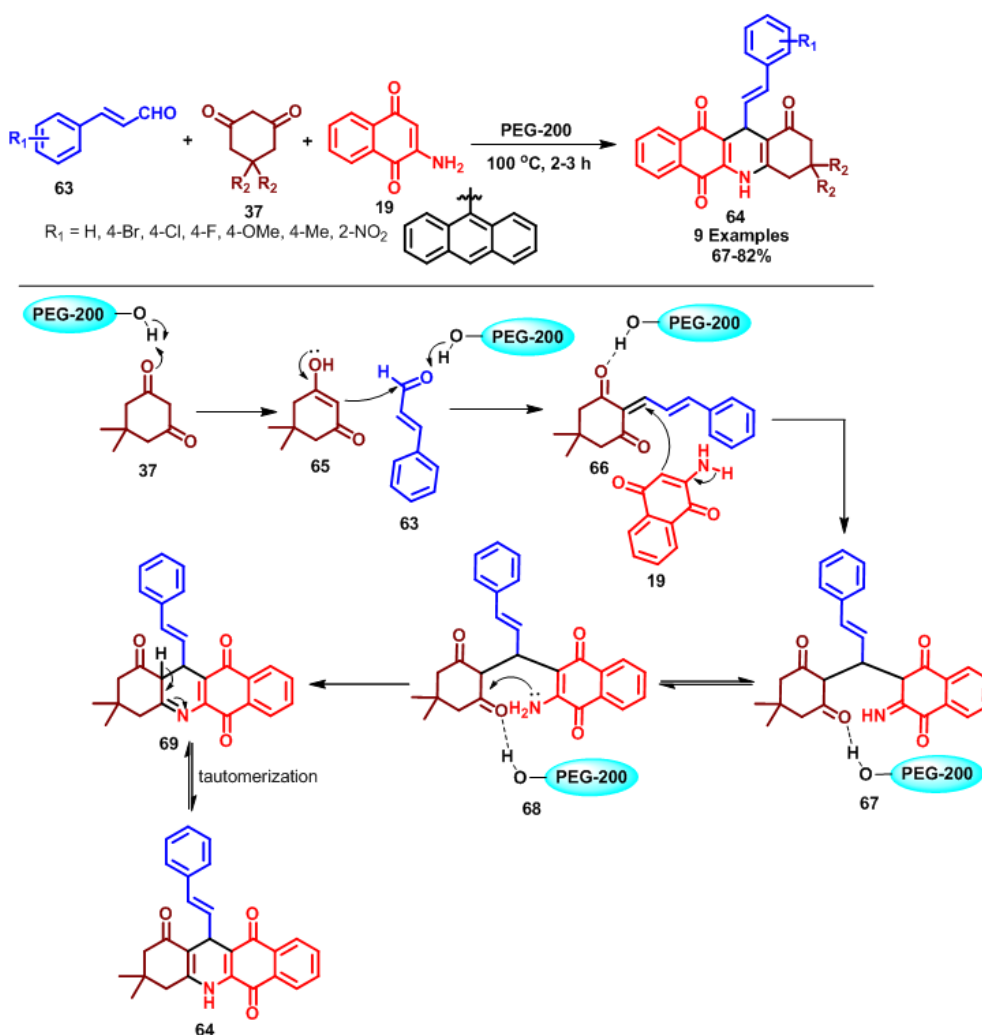
The tetronic acid [88] is an excellent scaffold for the synthesis of fused *N*-heterocycle. In recent years its use has been increased in the multicomponent reactions for the synthesis of diversely substituted and functionalized *N*-heterocycles. In this context, in 2022, Nguyen and co-workers reported a microwave mediated multicomponent approach for the synthesis of naphthoquinone-fused dihydropyridines 54 in presence of *p*-TsOH at 120 °C (Scheme 14) [89]. The reaction of 2-aminonaphthoquinones 19, tetronic acid 53 and aromatic and heteroaromatic aldehydes 10 in acidic medium result the formation of the naphthoquinone-fused dihydropyridines in 73-88% yields. The synthesized molecules were subjected to their biological evaluation and some of the compound showed good anticancer property. The mechanism proposed is shown in Scheme 16. The condensation of 2-aminonaphthoquinone and aldehyde forms the intermediate 59 which is attacked by the tetronic acid 53 in a conjugate manner to form the intermediate 60. Intramolecular cyclization followed by dehydration generated the final product 54.



Scheme 16. Acid mediated synthesis of naphthoquinone-fused dihydropyridines.

In a recent report, Mehar and Parvin reacted aminonaphthoquinone 19, dimedone/cyclohexane-1, 3-dione 37 and α , β -unsaturated aldehydes 63 in PEG-200 medium in catalyst-free conditions at 100 °C, and isolated styryl linked naphthoquinone fused dihydropyridines 64 in 67-82% yields (Scheme 17) [90]. This method was also applied for the gram scale synthesis of the products. No column chromatographic purification was needed to isolate the products. Other solvents such as H₂O, acetonitrile, MeOH, EtOH, PEG-400, glycerol, ethylene glycol was also screened during optimization study

in catalyst free condition. But PEG-200 was proved to be the best solvent for the synthesis of the products in the catalyst-200 is the better solvent than PEG-400 in terms of product yield and reaction time. As per the report, in presence of PEG-200, the aldehyde 63 and dimedone condenses to form the intermediate 66. Now the nucleophilic attack of 19 results in the formation of intermediate 67. On subsequent intramolecular cyclization and tautomerization the final product was generated.



Scheme 17. Multicomponent synthesis of styryl linked naphthoquinone-fused dihydropyridines.

4. Conclusion and Outlook

The medicinal dominance of naphthoquinone and naphthoquinone-fused *N*-heterocycles demands the development of new sustainable strategies to construct variable functionalized naphthoquinone-fused *N*-heterocycles. In this context, the 2-aminonaphthoquinone has been recognised as an effi-

cient candidate by the synthetic organic chemists. It shows 1, 3-C, N-binucleophilic property. Utilizing this property several MCRs have been developed in recent years. This review covers significant achievements in this area during 2019-2024. There are tremendous potentials in this field for further development. In case of the naphthoquinone-fused pyrroles very less number of 1, 3-dicarbonyl compounds have been explored and there is no report on the utilization of acyclic 1,

3-dicarbonyls. Similarly in case of naphthoquinone-fused dihydropyridines only the tetronic acid, dimedone and cyclohexane-1, 3-dione were explored. This molecule needs more attention for further advances in synthetic and medicinal chemistry. One major yet challenging aspect in this field has remained unaddressed. There is no report on the utilization of the acyclic 1, 3-dicarbonyls. There are several important acyclic 1, 3-dicarbonyls for example the naturally occurring and medicinally active curcumin that possess 1, 3-dicarbonyl functionality. They may be the important candidates in medicinal chemistry. More research is expected in this area in future.

Abbreviations

MCR	Multicomponent Reaction
DMSO	Dimethylsulfoxide
DMF	N, N-Dimethylformamide
DCE	1, 2-dichloroethane
FDA	Food and Drug Administration
HIV	Human Immunodeficiency Virus
HFIP	1, 1, 1, 3, 3, 3-hexafluoroisopropanol
NQ	Naphthoquinone
PTSA	<i>para</i> -Toluenesulfonic Acid
PEG	Polyethylene Glycol

Acknowledgments

The authors greatly acknowledge Berhampur University, Bhanja Bihar for providing infrastructure. Swadhin Swaraj Acharya thanks CSIR, New Delhi, India for the junior research fellowship. Dr. Parida is grateful to the funding agencies Mukhyamantri Research & Innovation (MRI)-OSHEC, Odisha (293/249/OSHEC), SERB (SRG/2019/002032), New Delhi, India, S & T Department Project-Odisha (ST-SCST-0061/2018/2724), UGC (F. 30-484/2019) (BSR) for generous funding to this work. The timely help of Dr. Arnab Chakraborty (IIT Patna), Subrat Sahu (IIT Bhubaneswar), Akash Bisoyi, Alisha Rani Tripathy (IISER Thiruvananthapuram) is greatly acknowledged.

Author Contributions

Swadhin Swaraj Acharya: Conceptualization, Investigation, Software, Writing – original draft, Writing – review & editing

Bishwajit Das: Software, Writing – original draft

Bibhuti Bhusan Parida: Conceptualization, Formal Analysis, Funding acquisition, Investigation, Project administration, Software, Supervision, Writing – review & editing

Data Availability Statement

Not applicable for this article because no data were re-

quired and analysed in this study.

Conflicts of Interest

The authors declare no conflicts of interest.

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