

## Biochemically Synthesized Potential Aminocoumarin-Glycoside Antibiotics

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### Abstract

Among the broad range of naturally occurring antibiotics, the aminocoumarin glycoside antibiotics have drawn immense attention due to their potential to inhibit the function of DNA gyrase, an essential bacterial enzyme, which is involved in controlling topological transition of DNA. However, the poor pharmacological properties of these molecules have always been a hindrance towards their widespread clinical application. Being equipped with genetic modification techniques, *Streptomyces* strains have been engineered to produce a diverse range of new aminocoumarin antibiotics, which are more active than their naturally occurring entities. In this review, we have compiled five aminocoumarin antibiotics i.e. novobiocin, clorobiocin, coumermycin A1, simocyclinone D8 and rubradirin, which have been produced through biochemical methodologies. Basic structure of this class of molecules contains aminocoumarin moiety, a sugar moiety and a substituted benzoic acid moiety/ pyrrole moiety/ angucyclinone moiety/ picolinic acid moiety. In novobiocin, clorobiocin and coumermycin A1, the sugar moieties are attached with the aminocoumarin through *O*-glycosidic bond, whereas in simocyclinone and rubradirin, the sugar moiety is attached with the additional structural moieties of these molecules.

**Keywords:** Aminocoumarin glycosides; Biochemical synthesis; Coumarin core; Antimicrobial agents.

### 1. Introduction

The genus *Streptomyces* consists of a gram-positive, aerobic and filamentous bacteria, which produce a variety of biologically active secondary metabolites. Along with antifungal, antiviral, antitumor, antihypertensive and immunosuppressive compounds,<sup>1,2</sup> *Streptomyces* species also produce almost two thirds of clinically used natural antibiotics.<sup>3</sup> The production of these secondary metabolites help to save different species of microorganisms of this genus from other pathogenic microorganisms. Sometimes through

the process of symbiosis, the antibiotic secondary metabolites of *Streptomyces* also protect plants against pathogens.<sup>4</sup> Starting from the discovery of antibiotics, like streptothricin and streptomycin in 1950s, a great number of antibiotics which have been used to treat infectious diseases in human are sourced from genus *Streptomyces*.<sup>5,6</sup> Genetic engineering studies and combinatorial biosynthetic methodologies revealed that genes responsible for the biosynthesis of these secondary metabolites are positioned as cluster at a specific location in the genome of these micro-

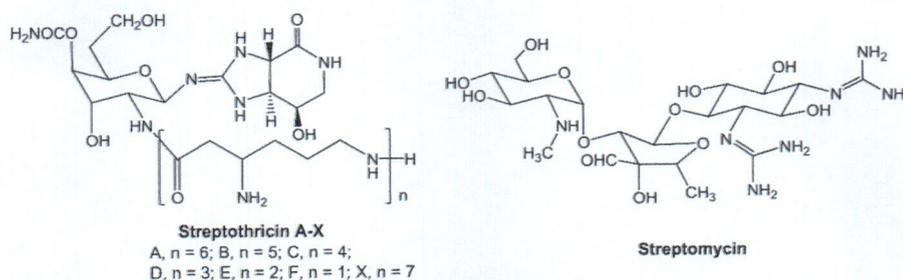


Figure 1. Streptothricin and streptomycin.

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## Multicomponent one pot synthesis of C-glucosides of 1-azaindolizines

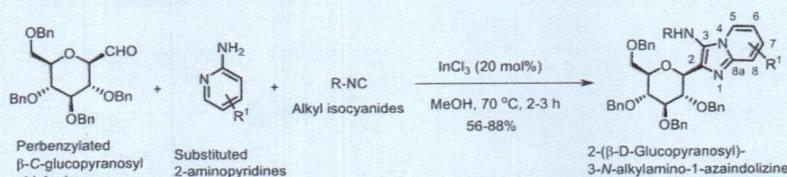
Banty Kumar<sup>a,b</sup>, Bhawani Shankar<sup>a</sup>, Sandeep Kumar<sup>a</sup>, Jyotirmoy Maity<sup>c</sup>, and Ashok K. Prasad<sup>a</sup>

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### ABSTRACT

A protocol based on Groebke-Blackburn-Bienayame (GBB) multicomponent reaction has been developed for efficient and atom economical synthesis of C-glucosides of 1-azaindolizine, i.e. 2-(β-D-glucopyranosyl)-3-N-alkylamino-1-azaindolizine. Thus, a series of fourteen novel 2-(β-D-glucopyranosyl)-3-N-alkylamino-1-azaindolizines have been synthesized in moderate to good yields by reaction of a perbenzylated β-C-glucopyranosyl aldehyde with differently substituted 2-aminopyridines and alkyl isocyanides using InCl<sub>3</sub> as acid catalyst. All synthesized β-C-glucosides were unambiguously characterized with the help of spectroscopic (IR, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and mass spectra) data analysis.

### GRAPHICAL ABSTRACT



### ARTICLE HISTORY

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### KEYWORDS

β-C-glucopyranosyl aldehyde; C-glucosides of 1-azaindolizines; 2-(β-D-glucopyranosyl)-3-N-alkylamino-1-azaindolizines; multicomponent reaction; perbenzylated

## Introduction

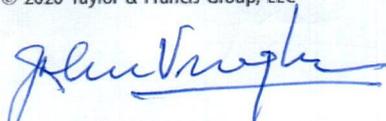
Heterocyclic amines are essential molecules of life. 1-Azaindolizine is one of those important heterocyclic structural moieties, which has drawn the attention of synthetic chemists in recent past due to its wide range of biological activities, such as antibacterial,<sup>[1]</sup> anti-inflammatory,<sup>[2]</sup> antimicrobial,<sup>[3]</sup> anticancer,<sup>[4]</sup> anti-HIV,<sup>[5]</sup> anti-protozoal<sup>[6]</sup> and anti-osteoporotic<sup>[7]</sup> effects. 1-Azaindolizine is structural motif of many drug molecules such as saripidem (anxiolytic), alpidem (anxiolytic) and zolpidem (hypnotic) (Figure 1). [8,9]

This structural moiety, i.e. 1-azaindolizine can be synthesized through tandem Michael addition reaction,<sup>[10]</sup> microwave assisted Domino's reaction,<sup>[11]</sup> oxidative coupling reaction<sup>[12]</sup> and electrocyclization reaction.<sup>[13]</sup> However, an elegant multicomponent reaction (MCR) always remains an excellent route to get this molecular scaffold.

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## Medicinal Chemistry &amp; Drug Discovery

Synthesis and Antitubercular Activity of 4,5-Disubstituted  $N^1$ -(5'-deoxythymidin-5'-yl)-1,2,3-triazolesRajesh Kumar,<sup>[a, b]</sup> Devla Bimal,<sup>[a]</sup> Kavita,<sup>[a]</sup> Manish Kumar,<sup>[a]</sup> Divya Mathur,<sup>[a]</sup> Jyotirmoy Maity,<sup>[c]</sup> Sunil K. Singh,<sup>[d]</sup> M. Thirumal,<sup>[a]</sup> and Ashok K. Prasad\*<sup>[a]</sup>

Synthesis of fifteen  $C^4$ -aroyl- $C^5$ -aryl- $N^1$ -(5'-deoxythymidin-5'-yl)-1,2,3-triazoles have been reported starting from azidation of 5'-*p*-toluenesulfonyloxythymidine followed by azide-alkene oxidative cycloaddition reaction of the resulted 5'-azido-5'-deoxythymidine with 1,3-diarylpropenones in dimethylformamide (DMF) in the presence of tetra-*n*-butylammonium hydrogen sulfate ( $n$ -Bu<sub>4</sub>N<sup>+</sup>HSO<sub>4</sub><sup>-</sup>, TBAHS) as catalyst in 60 to 79% overall yields. Further, they were also synthesized by one pot sequential reaction of tosylated thymidine with sodium azide in DMF and then with 1,3-diarylpropenones in presence of  $n$ -Bu<sub>4</sub>N<sup>+</sup>HSO<sub>4</sub><sup>-</sup> in superior yield of 70 to 95% than 60 to 79% in two step procedure. All fifteen synthesized compounds were screened for their *in vitro* anti *Mycobacterium tuberculosis* activity against

sensitive reference strain H37Rv and multi drug resistant (MDR) clinical isolate 591, and found to exhibit minimum inhibitory concentration (MIC) ranging from 2 to 15  $\mu$ g/mL, which was equivalent to the MIC of first line anti-tubercular drug streptomycin. All compounds qualify for their drug likeness when their physicochemical parameters were assessed using online MolSoft and Lipinski filter software, except their molecular weight. The cytotoxicity of potent compounds evaluated human monocytic cell line THP-1 by 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl-2*H*-tetrazolium bromide (MTT) assay was found to be less as compared to the first line drug, isoniazid.

## Introduction

Tuberculosis (TB), predominantly caused by *Mycobacterium tuberculosis* (*M. tuberculosis*) is one of the biggest health threat causing morbidity and mortality worldwide.<sup>[1]</sup> The current empiric therapy of combined use of four first-line anti-TB drugs (isoniazid, rifampicin, ethambutol and streptomycin) known as directly observed treatments (DOTS) launched by WHO requires 6–9 months of treatment and the cure rate is approximately 85%.<sup>[2]</sup> The situation of TB treatment has worsened with the emergence of new *M. tuberculosis* strains that are resistant to DOTS treatment. Cases of TB due to lethal drug-resistant strains (DR-TB), multidrug-resistant strains (MDR-TB) and extensively drug-resistant strain (XDR-TB) are on the rise, which has warranted discovery of new TB treatment regimens and

chemotherapeutic agents.<sup>[3–6]</sup> Design of nucleoside based drugs capable of interfering with *mycobacterial* purine and pyrimidine metabolic pathways is a promising approach towards the future of anti-TB therapy.<sup>[4–9]</sup> Two 5'-modified lead nucleoside antibiotics, SQ641<sup>[5]</sup> and CPZEN-45<sup>[6]</sup> are currently undergoing preclinical testing as an anti-TB agent (Figure 1).

Substituted 1,2,3-triazoles have been considered as a privileged heterocyclic moiety owing to their indomitable biological potential<sup>[7]</sup> and ability to be linked to different pharmacophores to generate an array of new hybrid molecules with improved efficacy.<sup>[8–10]</sup> Herein, we describe facile synthesis of  $C^4$ -aroyl- $C^5$ -aryl- $N^1$ -(5'-deoxythymidin-5'-yl)-1,2,3-triazoles using two procedures, one by azidation of 5'-*p*-toluenesulfonyloxythymidine followed by condensation of azidothymidine with 1,3-diarylpropenones and the other by one-pot sequential reaction of tosylated thymidine with sodium azide in DMF and then with 1,3-diarylpropenones in presence of  $n$ -Bu<sub>4</sub>N<sup>+</sup>HSO<sub>4</sub><sup>-</sup> as catalyst involving oxidative azide-olefin [3 + 2] cycloaddition (OAO) protocol in good to excellent yields. The synthesized 4,5-disubstituted  $N^1$ -(5'-deoxythymidin-5'-yl)-1,2,3-triazoles were evaluated for their *in vitro* anti-TB activity against sensitive reference strain H37Rv & MDR clinical isolate 591 and for cytotoxicity activity against human THP-1 cells by MTT assay.

## Results and Discussion

The nucleoside precursor of 4,5-disubstituted  $N^1$ -(5'-deoxythymidin-5'-yl)-1,2,3-triazoles, *i.e.* 5'-azido-5'-deoxythymidine 2 was synthesized following literature procedure by selective mono-tosylation of thymidine with tosyl chloride followed by azidation of resulted 5'-*p*-toluenesulfonyloxythymidine with

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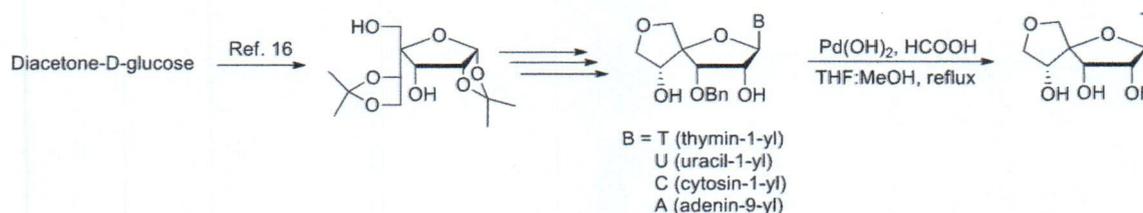
## Synthesis of conformationally restricted C-4'-spirofuranoribonucleosides

Neha Rana,<sup>a,d</sup> Manish Kumar,<sup>a,b</sup> Harbansh Singla,<sup>a</sup> Jyotirmoy Maity<sup>a,c</sup> and Ashok K. Prasad<sup>\*a</sup>

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### Abstract



The promising features of conformationally restricted C-4'-spiro-nucleosides led to the development of concise and efficient methodology for the synthesis of (1''R)-3'-benzyl-1''-hydroxy-5'-O,4'-C-ethylene-β-D-ribofuranosyl-nucleosides starting from diacetone-D-glucose in 20 to 28% yields. Thus, diacetone-D-glucose was converted to 4-C-hydroxymethyl-1,2:5,6-di-O-isopropylidene-β-D-gulofuranose following literature procedure, which was further converted to targeted conformationally restricted C-4'-spiro-nucleosides in five steps in 45 to 63% overall yields. Benzylated spiro-thymidine was converted to spiro-thymidine by affecting debenzilation with palladium hydroxide in THF:MeOH in the presence of formic acid and its S-type puckering was confirmed by X-ray crystallographic studies.

**Keywords:** Spiro-nucleosides, C-4'-spirofuranoribonucleosides, conformationally restricted nucleosides, S-type puckered spiro-nucleosides.

### 1. Introduction

In recent years, there has been an upsurge in the development of methodology for modifying the sugar moiety in numerous ways to synthesize conformationally constrained nucleoside analogues to improve their pharmaceutical properties.<sup>1-3</sup> In this connection, various research groups have synthesized different types of spiro-nucleosides containing spirocyclic ring, such as C-1'-spiro-,<sup>4,5</sup> C-2'-spiro-,<sup>6</sup> C-3'-spiro-<sup>7</sup> and C-4'-spiro-nucleosides (Figure 1).<sup>8</sup> Some of them have shown excellent antiviral activity.

In the year 1991, after the isolation of the naturally occurring nucleoside (+)-hydantocidin (5, Figure 1)<sup>9</sup> consisting of a spirocyclic ring at anomeric position, Paquette and coworkers<sup>10</sup> introduced the concept of spirocyclic restriction in nucleosides via insertion of a carbocyclic ring at C-4' of furanose rings that results in

the enhancement of biological activities of such compounds. In continuation Mandal and coworkers<sup>11</sup> synthesized four membered spiro-nucleosides at C-4' position, i.e. nucleosides 10 and Dang, *et al.*<sup>12</sup> synthesized a series of C-4'-spiro-nucleosides 11 which are potent inhibitors of HCV NS5B polymerase (Figure 1). Recently, our group has also carried out the synthesis of spirocyclic nucleosides consisting of a spirocyclic oxetane ring / tetrahydrofuran ring at their C4-position and have demonstrated the arrest of the sugar puckering of these nucleosides either in S-type or in N-type conformation.<sup>13</sup> The unprecedented structural features of C-4'-spirocyclic nucleosides 6-11<sup>14</sup> was found to restrict the conformation of the ribofuranose ring, permitting these nucleosides to acquire the optimal puckering required for specific biological activities (Figure 1).<sup>15</sup> Promising structural and biological features of C-4'-spiro-nucleosides inspired us to

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## Synthesis and Applications of Coumarin Glycosides: A Review

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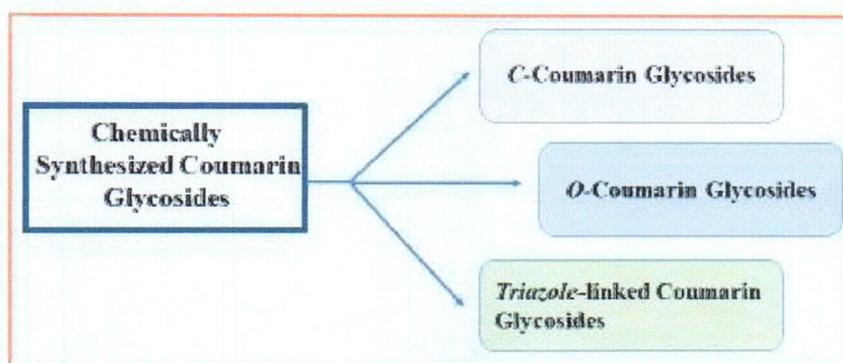
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### Abstract



Among naturally occurring biologically important molecules, coumarin glycosides constitute a significant class of organic compounds with huge medicinal importance. Exploration of potential of this class of molecules has shown their applicability as antibacterial, anticancer, anticoagulant agents. They were also found to be efficient enzyme inhibitors and fluorophores. In this review we have compiled the chemical methodologies for the synthesis of this class of molecules and discussed their varied applications.

**Keywords:** C-coumarin glycosides; O-coumarin glycosides; Triazole-linked coumarin glycosides.

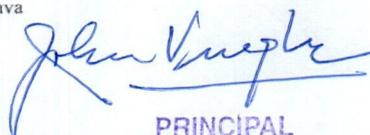
### 1. Introduction

Coumarins are heterocyclic natural products that consist of fused benzene and  $\alpha$ -pyrone ring. It was first isolated from tonka beans. These natural products possess a wide array of biological and pharmacological activity, which encouraged scientists and researchers around the world to synthesize their analogues by following numerous chemical methodologies. Coumarin moiety linked to a carbohydrate residue are called as coumarin glycosides. The molecules of this class of organic compound have shown an array of biological properties, such as antibacterial, anticancer, anticoagulant, enzyme inhibitors, and as well as displayed fluorescent properties.<sup>1</sup> When carbohydrates are coupled with small biologically active molecules it is envisaged that they will improve the pharmacological profile of those molecules without altering their activity and selectivity.<sup>2</sup> The sugar modules in the coumarin

glycosides contribute towards molecular recognition of their cellular target and help the aglycon part to bind with DNA strand.<sup>3</sup> Review articles on coumarins<sup>4,7</sup>, biscoumarins<sup>8</sup>, sugars<sup>9</sup> are abundant in literature but exclusive review on coumarin glycosides was not published for last two decades. Recently, we have discussed methodologies for synthesis of coumarin glycosides in a review article encompassing mutagenic synthesis, chemo-enzymatic synthesis and hairy root culture system synthesis.<sup>10</sup> However, the lack of an exclusive review article on chemically synthesized coumarin glycosides has encouraged us to compile the synthetic methodologies and their diverse applications in a well-organized manner for the convenience of the researchers working in this field.

Owing to diverse structural feature of the coumarin glycosides, we have categorized these molecules into three broad divisions, which are defined according to the attachment of the glycoside moiety with the aglycon

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# Synthesis of D-glycopyranosyl depsipeptides using Passerini reaction

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## ARTICLE INFO

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(2R)-2-(D-glycopyranosyl)-2-acyloxyacetamides and depsipeptides

## ABSTRACT

A protocol based on Passerini multi-component reaction has been developed for facile, efficient and atom economical synthesis of a small library of twenty potential bioactive (2R)-2-(D-glycopyranosyl)-2-acyloxyacetamides using perbenzylated D-glycopyranosyl aldehydes, substituted isocyanides and different aliphatic/aromatic carboxylic acids. All twenty synthesized D-glycopyranosyl  $\alpha$ -acyloxy amides, commonly known as depsipeptides were unambiguously identified on the basis of their spectral (IR, <sup>1</sup>H, <sup>13</sup>C NMR, COSY, HSQC, NOESY and HRMS) data analysis.

## 1. Introduction

The widespread use of multi-component reactions (MCRs) is associated with many advantages, such as one-pot synthesis, inherent simple procedure, time saving, the excellent bond forming ability, atom economy and waste minimization. Some examples of well-known MCRs are Hantzsch [1], Bignelli [2], Passerini [3] and Ugi [4] reactions, out of which Passerini reaction involving an isocyanide, an aldehyde and a carboxylic acid has been used for the synthesis of  $\alpha$ -acyloxy amides commonly known as depsipeptides [5–8].

Depsipeptides are biologically significant molecules possessing wide spectrum of activities, such as antibacterial [9], antiviral [10], anti-fungal [11], anti-inflammatory [12] etc. Natural products azinomycin A and B having  $\alpha$ -acyloxy amide moiety have been found to act as anti-tumor agents [13], valinomycin acts as an antibiotic [14], derivatives of 5-nitro-furan-2-carboxylic acid showed inhibition of RNase H function of HIV-1 reverse transcriptase [15] (Fig. 1). Depsipeptides have also been used as non-viral vectors for drug delivery showing higher tendency to penetrate biological membranes, which enables them to have high target occupancy and efficiency towards penetration into organs [16]. Further, few C-glycopeptidomimetic depsipeptides in which sugar aldehyde is attached to the anomeric position of the carbohydrate moiety and reverse C-glycopeptidomimetic depsides in which aldehyde group is located at the C-5 position of galactopyranosyl moiety has also been reported (Fig. 1) [5b,5c,5e].

Passerini reaction has also been used for the synthesis of numerous useful structural motifs, such as cyclic lipopeptides, which are antimicrobial natural products and exhibit wide spectrum of other biological activities (Fig. 1) [17–20]. These reports inspired us to synthesize sugar-based depsipeptides by the reaction of perbenzylated D-glycopyranosyl aldehydes with substituted isocyanides and different aliphatic/aromatic carboxylic acids in moderate to high yields.

## 2. Results and discussion

Synthesis of sugar precursors, C-(2,3,4,6-tetra-O-benzyl- $\beta$ -D-glucopyranosyl)formaldehyde (**1a**) and C-(2,3,4,6-tetra-O-benzyl- $\beta$ -D-mannopyranosyl)formaldehyde (**1b**) were carried out starting from native sugars, D-glucose and D-mannose, respectively in overall good yields following literature procedure [21]. For the synthesis of depsipeptides optimization of Passerini reaction condition was carried out to efficiently synthesize 2-(D-glycopyranosyl)-2-acyloxyacetamides. Thus, perbenzylated D-sugar aldehyde **1a** was reacted with 3-methoxy-2-methylbenzoic acid (**2a**) and cyclohexylisocyanide (**3a**) in different organic solvents, i.e. toluene, CHCl<sub>3</sub>, DCM, THF, EtOAc, dioxane, methanol, acetone, acetonitrile and DMSO (Table 1, entries 1–10). In the absence of any acid catalyst, DCM was found to be the best solvent to afford the desired depsipeptide, (2R)-2-(2',3',4',6'-tetra-O-benzyl- $\beta$ -D-glycopyranosyl)-2-acyloxyacetamide **4a** in 70% yield (Table 1, entry 3).

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# محسن کا کوروی کی قصیدہ نگاری

ڈاکٹر شمیم احمد

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ذرا ہٹ کر موسم بہار کا وہ لطف بیان کیا جائے جو خالص ہندوستانی رنگ کا حامل ہو۔ خالص ہندوستانی علامات ہوں۔ جملہ وفرات کے بجائے گنگا و جمنہ ہوں، سمرقند و بخارا کے بجائے مٹھرا اور کاشی بہار یہ تشبیہ کا موضوع بنیں۔ قصیدے کو بلکہ نعتیہ قصیدے کو خالص ہندوستانی رنگ عطا کرنے کی محسن کی کوشش نے انہیں قصیدہ نگاروں میں منفرد مقام عطا کیا ہے۔ ان کا مشہور زمانہ قصیدہ جس مطلع سے شروع ہوتا ہے اس کا آہنگ اور شکوہ ملاحظہ ہو:

سمت کاشی سے چلا جانے مٹھرا بادل  
برق کے کندھے پر لاتی ہے صبا گنگا جل

کاشی شیو دیوتا کی نگری ہے اور مٹھرا اثری کرشن کی۔ کاشی یعنی بنارس میں مقدس گنگا بہتی ہے جب کہ مٹھرا میں جمنہ اپنا زور دکھاتی ہے۔ شاعر کا تخیل کاشی سے مٹھرا کی طرف سفر کرنے والے بادلوں کو گنگا جل عطا کر دیتا ہے۔ گنگا کا یہ جل مقدس جل ہے، اب یہ جل مٹھرا میں جا کر بر سے گا اور جمنہ میں مل جائے گا۔ اس طرح گنگا اور جمنہ کا ملن کا رخا نہ قدرت سے ملے پائے گا۔ یہی ملن ہندوؤں اور مسلمانوں کی تہذیب کا ملن ہے جسے عرف عام میں گنگا جمنی تہذیب کہتے ہیں۔

اس ایک شعر سے جو پیکر ابھرتے ہیں وہ بھی غور طلب ہیں۔ بادل کا یہ پیکر اپنی جگہ ساکت و جامد نہیں ہے۔ ٹھہرا ہوا بادل نہیں ہے۔ یہ رواں ہے۔ چل رہا ہے، گویا چلتا ہوا بادل زندگی کی علامت ہے۔ غور کیجئے تو یہ بادل تبخیر کے عمل سے گزر رہا ہے اور صبا سے گنگا جل فراہم کر رہی ہے۔ یہ سب چلتے پھرتے رواں دواں پیکر ہیں۔ یہ اپنی روانی سے نظم کی ابتدا ہی میں ڈرامائیت کا عنصر پیدا کر رہے ہیں۔ ایک ہی شعر میں کئی کردار ہیں اور سب کسی نہ کسی عمل سے گزر رہے ہیں یا پھر کچھ کر کے دکھا رہے ہیں جو کہ ڈرامے کی اصل ہے۔ اسی ضمن میں غالب کے قصیدے کا وہ مطلع بھی ملاحظہ کیجئے جس میں انھوں نے فطرت کے اہم مظہر چاند کو موضوع بنا کر براہ راست ڈرامائی انداز پیدا کیا ہے اور چاند سے راست سوال کیا ہے:

ہاں مہ نو! سنیں ہم اس کا نام  
جس کو جھک کے کر رہا ہے سلام

فطرت کے مظاہر بھی عجیب و غریب ہیں۔ یہ ہر دن اپنی نئی شان دکھاتے ہیں اور ہر شخص کے لیے ان میں متاثر ہونے کی الگ الگ خصوصیات پائی جاتی ہیں۔ آسمان پر اڑتے بادل ہم نے بھی دیکھے ہیں۔ اس عام مشاہدے کو اپنے غور و فکر اور تخیل سے ایک خصوصی مشاہدے یا خصوصی تجربے میں بدل دینے کا ہر ایک حساس فنکار کو بہ خوبی آتا ہے۔ محسن کا کوروی ہمارے ایسے ہی فنکار ہیں جو مظاہر فطرت کو زندگی عطا کر دیتے ہیں۔ یہ مظاہر پڑھنے والوں کی آنکھوں کے سامنے چلتے پھرتے اور اپنے اپنے کردار اور اپنی اپنی خصوصیات کے بہ موجب حرکت کرتے ہوئے نظر آتے ہیں۔

اردو ادب میں محسن کی شناخت ایک قصیدہ گو کی حیثیت سے ہے۔ انھوں نے بیشتر قصیدے اس حضرت کی شان میں کہے ہیں یعنی زیادہ تر قصیدے نعتیہ قصیدے ہیں۔ محسن جیسے بڑے فنکار کو اس بات کا بہ خوبی علم ہوتا ہے کہ اس کے قلم سے جو کچھ جھپٹے تحریر میں آ رہا ہے اس کی نوعیت کیا ہے۔ وہ خالص نعت بھی کہہ سکتے تھے، یقیناً نعتیہ قصیدہ لکھنے والا آں حضرت سے خصوصی محبت کا حامل ہوگا، اس نے سیرت پاک کا بہ غور مطالعہ کیا ہوگا اور خصوصی جذبات و احساسات ہی نے اسے آمادہ کیا ہوگا کہ وہ اپنے جذبوں کا اظہار کرے، لیکن محسن کے قلم سے اگر یہ اظہار صرف نعت تک محدود ہوتا تو شاید ن کار کی روح کو تسکین حاصل نہ ہوتی۔ اسے تو اپنے فن کا اظہار بھی کرنا تھا۔ اسے تو یہ ظاہر کرنا مقصود تھا کہ وہ نعت رسول کے ساتھ قصیدے کے جز و لازم تشبیہ کے اشعار کہنے پر بھی بے پناہ قدرت رکھتا ہے۔ وہ نعت کے بجائے نعتیہ قصیدہ اس لیے لکھتا ہے تا کہ وہ دوران قصیدہ ایک غزل شامل کر کے اپنی فنکارانہ دست گاہ کا دعویٰ پیش کر سکے۔ آپ جانتے ہیں کہ انیسویں صدی کے نصف آخر تک شعری کمالات کے اظہار کے لیے قصیدہ کہنا ضروری سمجھا جاتا تھا۔ محسن تو منہ کا ذائقہ بدلنے کے لیے بھی دوسری اصناف سخن کی طرف توجہ نہیں کرتے وہ تو صرف قصیدے کہتے ہیں یا مثنویاں یا پھر رباعیات۔ غزل کی لطافت قصیدے کے ابتدائی اشعار میں تشبیہ کی صورت از خود چلی آئے گی۔

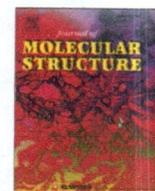
تشبیہ کی روایت کو حیات نوعطا کرتے ہوئے محسن نے موسم بہار کو موضوع بنا کر تہذیبیہ اشعار کہے ہیں۔ یہ کوشش بھی کی ہے کہ عام روایت سے

ایوان اردو، دہلی

دسمبر ۲۰۲۰

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# Experimental and computational investigation of polymorphism in methyl 3-hydroxy-4-(piperidin-1-ylmethyl)-2-naphthoate

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## ABSTRACT

A piperidine substituted methyl 3-hydroxy-2-naphthoate was synthesized for application as a supramolecular host, which yielded colorless and light yellowish orange crystals using different solvents for crystallization. The crystals of the substituted methyl 3-hydroxy-2-naphthoate were analysed using melting point, IR, Reflectance UV–Visible, fluorescence, SEM, <sup>1</sup>H NMR, DSC, PXRD and single crystal X-ray crystallographic techniques to reveal polymorphism. The crystal data were also analysed computationally using Gaussian 09, CLP-PIXEL, Crystal Explorer software to reveal the differences in the intermolecular interactions and optical properties. The short intermolecular interactions such as C–H ⋯ π and π ⋯ π interactions differentiated the polymorphs of the molecule studied here.

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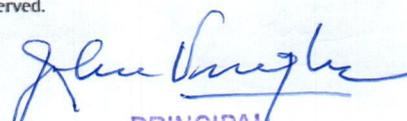
## 1. Introduction

Heterocyclic compounds have been investigated extensively for their applications in sensor development, pharmaceuticals, corrosion inhibitors, copolymers, dyes, veterinary products, solar cells and plant regulation [1–8]. In the area of pharmaceuticals, the bioavailability and dissolution rate may be affected by the crystallization of a molecule in different arrangements in the solid state [9–11]. The formation of two different sets of crystal creates either conformational isomerism or polymorphism [12]. Apart from pharmaceuticals, polymorphism also affects industries such as pigment, personal care, electronic, semiconductor, food and explosive industries [9]. The scientific term “polymorphism” is ubiquitous in the chemical sphere today, largely due to the unique properties exhibited by the chemically similar but altered molecules [12]. Crystal engineering has garnered huge interest in supramolecular chemistry owing to the structural variation at the individual atomic or molecular packing level [13–15]. It is quite feasible on the part of the molecular units or subunits to show such variations in response to temperature, pressure, solvents etc. [16,17]. Such structural variations at supramolecular level are scientifically termed as “polymorphs” [16,18]. Generally,

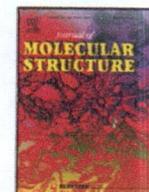
polymorphs provide their own uniqueness to the overall structural features in terms of shape, structure and geometry. The unique properties associated with the polymorphic forms are responsible for the rapid growth of crystal polymorphism across the disciplines of the chemical and pharmaceutical industry [19]. Normally, polymorphs differ in color, crystal parameter, crystal packing or lattice energy [20]. The design of molecular topologies and crystal packing plays an important role with respect to stabilization and engineering [21]. The existence of non-covalent interactions in the polymorphic crystals gives rise to unique physical, chemical properties, solubility and hygroscopic behaviour [22]. The diversity of intermolecular interactions provide significant information regarding supramolecular spacing and growth [23,24]. Polymorphism can also be induced through the use of different stimuli such as solvent, which provide an easy route to the polymorphism or pseudo-polymorphism in the crystals [25,26]. Various approaches for the crystal structure determination of the polymorphs are available, which include: X-Ray investigation [27], DFT methods [8,28,29], CLP-PIXEL for energy estimation [30], DSC, TGA, DTA for thermal stability [8,31,32], Crystal explorer program for surface and intermolecular interaction analysis [8,33], UV–visible, fluorescence spectroscopy for optical studies [34] and microscopy [35–37]. In the area of sensor development, the pre-organization of the receptor molecule is vital, which may also influence the optical properties [34]. The change in optical properties of the supramolecular device in the solid state is extremely important in the area of

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# A MC-spiropyran for smartphone assisted reversible, selective and nanomolar level detection of formic acid in water and gas phase

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## ABSTRACT

A *p*-toluenesulfonate salt of benzothiazonium (merocyanine) dye substituted with diethylamine was synthesized to act as an optical probe. The probe was characterized using FT-IR, NMR, HR-MS and examined for a response towards polar molecular and ionic species using UV-Visible, fluorescence spectroscopic techniques. The probe displayed remarkable formic acid-responsive optical and aggregation properties in distilled water and at pH 7.6 (1.0 mM HEPES). The change in color was also exploited for smartphone assisted digital colorimetric analysis of formic acid and formate ions. The formic acid response can be switched off thermally. The emission response of the probe appeared in the visible region. The receptor can also be used to detect the vapors of formic acid reversibly in air. The computational calculations based on M06-2X/6-31+G(d), M06-2X/6-31+G(d,p) and CAM-B3LYP/6-31+G(d,p) methods were performed to investigate the complex formation at a molecular level. The time-dependent CAM-B3LYP/6-31+G(d,p) method-based calculations were also implemented to study the observed absorption spectra of the complex.

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## 1. Introduction

Formic acid is the simplest carboxylic acid derivative, which is extremely important owing to its practical utilization in industries, agriculture, and biological systems [1–5]. In smaller concentrations, formic acid can be utilized as a food preservative and is frequently added to animal food and silage, owing to its antibacterial properties [6,7]. It also helps in fermentation [8]. For the same antibacterial property, it is used in agriculture around the world [8]. Formic acid is also produced in small quantities in our bodies from methanol, which we either consume, inhale or produce through biological processes [9]. It is also present in bees and ants' stings or bites [10,11]. It can be converted to H<sub>2</sub> and CO<sub>2</sub> under mild conditions [1,12]. The CO<sub>2</sub> produced in this process can be recycled into carbon-neutral fuel cycle [1,12]. Therefore, formic acid can potentially be used for the storage of hydrogen gas [1,12]. A formic acid powered transportation is planned for future, which is based on the formic acid-hydrogen fuel cycle [13]. The chemical precursors of formic acid mainly comprise acetylene, monoterpenes, and alkenes [14]. Besides terrestrial vegetation, biomass burning, as well as combustion, also contributes to the major source of formic acid [15]. Extensive utilization of

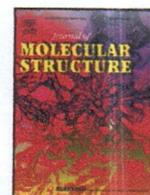
formic acid may also lead to diffusion of acidic substances into the atmosphere that may cause rainwater acidity [16] and hence plays an important role in atmospheric chemistry [17]. It also affects aqueous phase chemistry, as many reactions that occur in the environment are highly prone to pH, which ultimately depends on formic acid distribution [18]. A lower concentration of formic acid may be unnoticeable or marginally irritating to humans, but its higher concentration may lead to adverse effects, mainly due to its corrosive action. These include burns, ulcers, nausea, and blisters. Formic acid exposure also creates toxicity due to its inhibiting tendency towards mitochondrial cytochrome oxidase [19]. Due to the above stated harmful effects and utility of formic acid, monitoring its concentration is extremely essential. The reported detection methods for formic acid involve chromatographic methods [20,21], microwave technique [22] and electrochemical methods [23]. The direct detection of formic acid through optical signal can be a relatively easy, affordable and convenient method, which is applicable under a variety of field conditions. In order to achieve the desired objective, the spiropyran based materials are useful devices, due to their stimuli responsive properties. Spiropyran based materials have triggered much attention as sensing devices owing to their extreme flexibility, reversible nature and direct mode of action [24–26]. Spiropyran based materials are known to exist in colorless spiro form and colored merocyanine forms. The merocyanine forms display a tendency to form aggregates by virtue of their planar and highly conjugated structure. The intriguing process of formation

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# Palladium induced activation of a substituted naphthopyran for smartphone assisted sensitive and selective sensing in aqueous solution

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## ABSTRACT

A substituted naphthopyran moiety was synthesized to act as a probe for metal ions. Structural characterization techniques such as IR, NMR, HRMS, and single-crystal X-ray crystallography were used to establish the structure of the light-responsive photochromic probe. The intermolecular interactions were quantitatively investigated using the crystal explorer program. The affinity of the naphthopyran derivative as a probe for metal ion was investigated using different metal ions in a buffered medium to avoid the effect of change in pH during the recognition process. A bathochromic shift in the absorption band from ~354 nm to ~365 nm (broad band extended up to 450 nm) was observed upon addition of 10 equivalents of palladium ions. The smartphone assisted sensing of palladium ion based on digital images provided an excellent value of LOD (0.69  $\mu$ M), which was better than the LOD value obtained using UV-visible spectroscopy. The process of complex formation between the probe and the palladium ions was also investigated using NMR and computational methods. The computational methods were used to investigate the effect of water on the stability of the complex geometries. Furthermore, time-dependent DFT calculations were carried out to investigate the UV-Visible absorption spectra observed experimentally for the probe and the complex.

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## 1. Introduction

Metals like palladium (Pd) draws attention around the world owing to its impressive catalytic functions in profitable petroleum and chemical industries [1–7]. Palladium based catalysts are widely used in several well-known reactions due to high selectivity and activity associated with it that helps it to efficiently catalyze reactions like Heck, Suzuki-Miyaura, Sonogashira, Buchwald-Hartwig reactions, etc. [1,2,8–11]. In daily life, palladium is widely used in common devices like spark plug, electrical contacts, dentistry, catalytic converters for vehicles, etc. [5,6,12–16]. Besides, palladium in the form of palladium and silver alloy is used in hydrogen purification due to its ability to allow only monoatomic hydrogen to pass through it above 300 °C [17]. Palladium metal can absorb a large volumetric quantity of hydrogen gas at its surface and facilitates its reaction with oxygen or any other oxidizing agent through an electrochemical reaction, due to which it finds application in fuel cell [18]. Large scale utilization of palladium in chemical and petroleum industries is causing excessive release of waste material

loaded with it in our water bodies [13,19]. The detrimental effect of palladium originates from its ability to coordinate thiol or thioether groups of amino acids, proteins, DNA and vitamin B6 in biological systems [20–23]. The toxic nature of palladium is a cause of several diseases like an irritation to eye, skin, breathing tract, and even cancer-producing fatalities [13,24–27]. World health organization investigated the effect of the presence of palladium metal ion in water and confirmed the deadly dose to be 5–10 ppm and recommended control of daily uptake at ~1.5 to 15  $\mu$ g/day [28–31]. The wide utility of palladium and its toxic effect necessitates the development of new protocols for monitoring the concentration of this metal in our environment. Methods based on instrumental techniques like atomic absorption spectroscopy (AAS) [32,33], UV-Visible Spectroscopy [19,34,35], fluorescence spectroscopy [19,36–38], X-ray Powder Diffraction (PXRD) technique [39], high performance liquid gel chromatography (HPLC) [40], polymer-based sensor [38,41] etc. are generally utilized for the detection and determination of palladium ions. Most of the instrumental methods are bulky, expensive, and require sample collection and treatment at selected sites. Among them, colorimetric methods employing synthetic molecular receptors represent the fast, simplest, cheap, selective, sensitive, and portable methods for the detection of

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## Organic &amp; Supramolecular Chemistry

## Cyanide-Ion-Induced J-Aggregation of Merocyanine Dye for Paper-Based Colorimetric Detection in Water

Arvind Kumar,<sup>[a]</sup> Rajesh Kumar,<sup>[b]</sup> and Satish Kumar\*<sup>[a]</sup>

A probe based on merocyanine dye was synthesized as a salt of *p*-toluenesulfonate. Owing to the high solubility of merocyanine dye in water and H-bonding ability, it was investigated for anion-induced aggregation properties in the buffered medium [CH<sub>3</sub>CN: water (1:1/v: v)]. The probe recognized the existence of cyanide ion by forming an H-bond and displacing the *p*-toluenesulfonate group. The probe displayed a bluish-purple color in response to cyanide ion addition, which was initially light pink. The complex formation and aggregation properties of the probe were investigated using techniques like naked eye

detection, digital colorimetry, UV-Visible, <sup>1</sup>H-NMR, and dynamic light scattering (DLS). Paper and solution-based techniques aided by a smartphone were also used successfully in the determination process. An excellent limit of detection value (LOD) was witnessed in all the techniques exploited for sensing cyanide ions. Methods based on density functional theory (DFT) were also used to investigate the complex formation and observed a good correlation in experimental and theoretical results.

## Introduction

Cyanide ion is widely recognized for its lethal effects on humans, animals, as well as aquatic life.<sup>[1]</sup> The accidental exposure to cyanide ion led to many reported lethal accidents, which caused immense damage to the human population throughout the world.<sup>[2]</sup> The high concentration of cyanide in seawater causes inhibition in the reproduction of many species of fish and reduces their swimming ability.<sup>[3]</sup> Besides, cyanide poisoning also affects the birds<sup>[4]</sup> and mammal life.<sup>[5]</sup> The cyanide ion in nature arises through sources like apricot kernels, cassava roots, apple seeds, and bamboo shoots.<sup>[6]</sup> The levels of cyanide ion in the environment multiply due to human activities like the use of cyanide in paper, textile and plastic industries.<sup>[3b]</sup> Cyanide salts are also used in metallurgy for extraction and purification of gold,<sup>[7]</sup> in photography,<sup>[8]</sup> and the eradication of blights in ships and buildings.<sup>[9]</sup> Cyanide poisoning can be caused by inhaling hydrogen cyanide gas<sup>[10]</sup> or intake of salts of cyanide ion.<sup>[11]</sup> Although, the human body can detoxify small amounts of cyanide present in apple seeds and tolerate cigarette smoking,<sup>[12]</sup> an overdose of this ion causes poisoning.<sup>[13]</sup> Symptoms of cyanide poisoning include vomiting, fast heart rate,<sup>[14]</sup> cardiac arrest, shortness of breath,<sup>[15]</sup> and low blood pressure.<sup>[16]</sup> Cyanide ion normally prevents the

functioning of cytochrome C oxidase by inhibiting electron transport to oxygen during aerobic cellular respiration as a result of iron-cyanide ion complex formation in the mitochondria of the cell.<sup>[17]</sup> The inhibition in the electron transport chain ultimately leads to cell damage, and finally death.<sup>[17]</sup>

Owing to the highly toxic nature of cyanide ions, there is a growing interest in the research community toward monitoring its concentration in water bodies. During the last few decades, numerous experiments have been carried out to identify the existence of cyanide ions in drinking water<sup>[18]</sup> and eatables.<sup>[19]</sup> One of the most common methods to monitor the existence of cyanide ions is the use of chemical probes.<sup>[20]</sup> Several designs and scaffolds have been synthesized to detect or determine cyanide ions in solution. Techniques like UV-Visible spectroscopy,<sup>[21]</sup> fluorescence spectroscopy,<sup>[22]</sup> atomic absorption spectroscopy,<sup>[23]</sup> ion-exchange chromatography,<sup>[24]</sup> etc. have been employed extensively to study the interaction between different types of probes and cyanide ion in several types of medium. Among these, visible change of color is the most convenient method for the detection of cyanide ions. Different types of colorimetric reagents perceive cyanide ions presence in solution are available.<sup>[25]</sup> Probes for cyanide ions are either based on a chemodosimeter approach or complex formation through non-covalent interactions.<sup>[25b]</sup> Formation of J- or H- aggregate is also an interesting and intriguing approach for the development of a colorimetric probe for cyanide ion due to an abrupt change in the color of the solution.<sup>[26]</sup> An alteration in the color of the solution brought exclusively by the interaction of a cyanide ion with the groups present in the probe can be used to mark its presence in the solution. An analyte like cyanide ion causes either a disruption in the aggregation state of the probe or produces an aggregated structure through the influence of the intermolecular interactions. Also, the use of an aqueous solution unlike organic solvent is beneficial in terms of the utility of the probe

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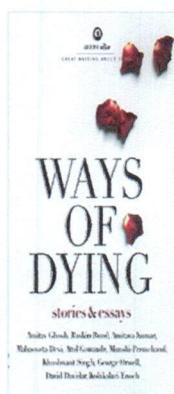
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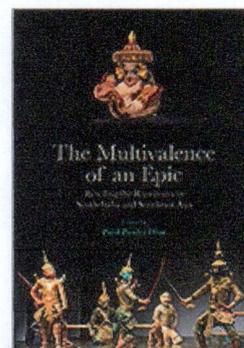
WAYS OF DYING: STORIES & ESSAYS by Amitav Ghosh, Ruskin Bond, Amitava Kumar, Mahasweta Devi, Atul Gawande, Munshi Premchand, Khushwant Singh, George Orwell, David Davidar, Kolakaluri Enoch  
Aleph Book Company, 2020, 124 pp., 399.00

JANUARY 2021, VOLUME 45, NO 1

Ways of Dying: Stories & Essays is the sixth publication in the Aleph Olio series. Much like other works in the series such as *Love and Lust*, *Notes from Hinterland*, *In a Violent Land*, *Ways of Dying* is an 'olio' or miscellany of remarkable works of fiction and non-fiction, all of which harp on the sure companion of life: death.

Through the assemblage of works from renowned writers such as Amitav Ghosh, Mahasweta Devi, Ruskin Bond, George Orwell, Kolakaluri Enoch and others, the anthology traverses across multiple spatio-temporal geographies, with a few works rendered in translation. Through this, the collection explores not only the

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TRIBUTE



## A Palanpur Chronicle

**Ann Susan Aleyas**

**RUMBLE IN A VILLAGE: A NOVEL** by Luc Leruth with Jean Drèze *Aleph Book Company, 2020, 408 pp., 699.00*

FEBRUARY 2021, VOLUME 45, NO 2

Rumble in a Village is an entertaining addition to literary representations of twentieth century rural India. In some ways reminiscent of Sri Lal Shukla's *Raag Darbari*, this novel is a collaborative effort of the Belgian born Indian economist Jean Drèze, and his friend and writer Luc Leruth. While Leruth has previously authored fiction set in India, this would be Jean Drèze's first in the field, a departure from his previous publications on public policy and developmental economics. As the Preface of the novel introduces to the reader, *Rumble in a Village* is a blend of the personal, historical and the imaginative, as it fictionally animates Jean Drèze's notes from the 1980s, which were made during his research work in Uttar Pradesh. The novel, as its title suggests, is a delightfully organized chaos of various intertwined lives in the village of Palanpur in western Uttar Pradesh. Though the narrative sets itself as a murder mystery, this trajectory is effortlessly interrupted, if not displaced, by the witty, light-hearted and realistic observations of the British Indian, Anil Singh.

*Rumble in a Village* traces the journey and experiences of Anil Singh, a banker living in London in the 1980s, following an unexpected call from India informing him of the death of

Palanpur. What awaits Anil in the village however are intricate webs of social relations, unforeseen encounters and commonly held secrets.

Aijaz Ahmad  
(1941–2022)

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# Convergence and global stability analysis of fractional delay block boundary value methods for fractional differential equations with delay

Surendra Kumar <sup>a</sup>✉, Abhishek Sharma <sup>a</sup>✉, Harendra Pal Singh <sup>b</sup>✉

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## Abstract

In this paper, a new numerical scheme termed as fractional delay block boundary value methods (FDBBVMs) is proposed. It is an extended version of the block boundary value methods (BBVMs). The proposed scheme is used to find numerical solutions of the fractional delay differential equations including Caputo fractional derivative of  $\beta^{\text{th}}$  order with  $0 < \beta < 1$  and a constant delay term. The estimation of the fractional-order derivative term is obtained by combining the  $m^{\text{th}}$ -order Lagrange interpolating polynomial along with the  $p^{\text{th}}$ -order BBVMs, and the constant delay term is dealt with certain modifications in the BBVM. Further, the convergence analysis of the proposed scheme is discussed and it is observed that the FDBBVM is convergent with order  $\min\{p, m - \beta + 1\}$ . Moreover, the scheme is shown to be globally stable and its computational efficiency and accuracy has been illustrated with the help of numerical examples.

## Introduction

In real life problems, differential equations play a prominent role in many disciplines such as

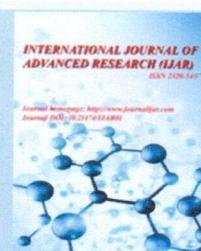
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## RESEARCH ARTICLE

### ECO-FRIENDLY, COST EFFECTIVE, AUTO-STERILIZATION OF ISOLATION GOWN: RECENT AND FORTHCOMING ASPECT

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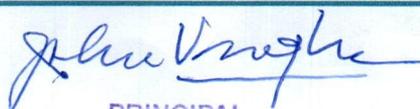
#### Abstract

This review addresses the issue of biomedical waste in the form of PPE (personal protection equipment) kit. COVID -19 pandemic has brought life into standstill. It has affected life of each and every person. This includes both direct effect in terms of health and indirect effect in terms of economic loss, business losses which have also resulted into sudden surge in unemployment. But apart from these directly visible effects there is one particular effect which is far sighted but cruel reality of present condition. It is the problem of biomedical wastage arising due to extensive use of PPE kits especially in the form of face mask and isolation gown. As layman is expected to wear face mask all the time, many countries has made it mandatory. It is very important to think about its effective, environment friendly disposal. Similarly isolation gown has also become very common in many medical and non-medical industries. If we don't dispose off used gown properly then the whole purpose of its use will get defeated as it further leads to disease transmission. While selecting method of disposal, it is very important to consider its adverse effect on environment. Incineration, shredding and deep pit burial are some of the common method of disposal of biomedical waste approved by health authorities of different countries, but these are not environment friendly. There are some other disinfection methods like ultra-violet (UV) irradiation, chemical disinfection with the help of them medical isolation gown can be reused but these methods have many disadvantages including being expensive, non-eco-friendly, some methods require expertise in handling machine, some affect the virus barrier ability of gown etc. Considering these problems, this review explored the idea of reusable isolation gown which doesn't require any expertise for its disinfection at the same time it doesn't adversely affect the environment. We proposed the idea of applying layer of photocatalyst which can inhibit the enzymatic activity of cells of microbes which will stop their replication. This can prove very useful in preventing the disease transmission. There are many reports on antimicrobial properties of various semiconductor photocatalyst which can further be improved by doping it with metal like Cu or Ag which are already popular for their medicinal properties. Major problem with most of the semiconductor

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# DEFAMILIARIZING THE FAMILIAL: A COSMOPOLITAN READING OF SATYAJIT RAY'S "AGANTUK"

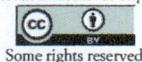
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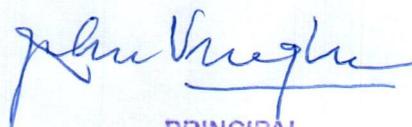
*Abstract:* In their introduction to the anthology *Cosmopolitanisms*, Breckenridge et al. describe cosmopolitanism as "ways of living at home abroad or abroad at home" (2000: 587). Cosmopolitanism, in these two dimensions, is enacted in Satyajit Ray's film *Agantuk* (1991) as well. While the dominant tendency in the film's reception has been to draw a dichotomy between parochialism and cosmopolitanism – with each proclivity identified with a different branch of the same family tree – this paper shall attempt to problematize this binary. Rather than articulating a tension between the home and the world, this paper proposes that *Agantuk* illustrates two different cosmopolitanisms – a way of "living at home abroad" and a way of "living abroad at home". While both cosmopolitan approaches diverge significantly, the film makes a strong case that they emanate from a common space of middle-class privilege and access, by contextualizing them against the economic liberalization reforms of 1991 India. Globalization is seen as fostering a banal, consumerist variety of cosmopolitanism – a means for a financially stable middle class to garner cultural capital, and to produce itself as "modern" on a global scale. It is this consumption-oriented cosmopolitanism that bears the brunt of the film's critical as well as recuperative efforts. Melted and recast, it has the potential to produce a "thicker", more inclusive form of local, everyday cosmopolitanism – a cosmopolitanism that is equipped to resist the impulse to flatten and commodify alterity, and to open itself to plural, co-existing modes of inhabiting modernity.

*Keywords:* Satyajit Ray, globalization in India, cosmopolitanism of consumption, middle class, modernity.

In their introduction to the anthology *Cosmopolitanisms*, Breckenridge et al. describe cosmopolitanism as "ways of living at home abroad or abroad at home" (2000: 587). Cosmopolitanism, in these two dimensions, is enacted in Satyajit Ray's film *Agantuk* (1991) as well. While the dominant tendency in the film's reception

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