SHIV NADAR
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INTER-INSTITUTION

COLLABORATION GRANTS

Title - Exploring Flexible UV LED for Disinfection of Secondary Treated Municipal Wastewater

UNDERTAKING FORM

Grant Title: Exploring Flexible UV LED for Disinfection of Secondary Treated Municipal Wastewater

Collaborating SNF Institutions:

We, Dr. Debajyoti Biswas, representing Shiv Nadar University Chennai and Dr. Tanusree Sengupta, representing SSN Engineering College, hereby acknowledge our joint collaboration in the submission of this grant proposal.

Principal Investigator (PI) Nomination for Communication:

In the spirit of effective communication and coordination, we nominate the following individual as the Principal Investigator (PI) responsible for primary communication on behalf of both institutions:

Primary PI Nominee:

Name: Dr. Debajyoti Biswas

Position: Assistant professor, Department of Computer Science and Engineering

Email: debajyotib@snuchennai.edu.in

Phone: 9176338192

Institution Name: Shiv Nadar University Chennai

Undertaking:

1. **Communication Responsibility:** The nominated Principal Investigator shall be responsible for all official communications related to the grant proposal, including but not limited to correspondence with the funding, updates, and reporting.

2. **Collaborative Decision-Making:** Both SNF Institutions commit to collaborative decision-making throughoutthe project duration. Major decisions, changes, or concerns will be discussed and agreed upon jointly, taking into consideration the best interests of the project and its objectives.

3. **Regular Updates:** The nominated Principal Investigator will provide regular updates to both institutions including the project review that shall take place every 6 months to assess progress and resolve any challenges, ensuring transparency and inclusivity in the decision-making process. Updates will be shared through agreed-upon communication channels.

4. **Amendment of PI Nominee:** In the event of unforeseen circumstances that require the amendment of the nominated Principal Investigator, Principal Investigator of other Institution will by default act as Primary Investigator for communication. Meanwhile, the Principal Investigator of other institution can be finalized mutually by both institutions timely and formalize the changes.

Signatures:

[Authorized Signature-

Dr. Debajyoti Biswas

Shiv Nadar University Chennai

Date: 17th December 2023

[Authorized Signature-

Dr. Tanusree Sengupta

SSN Engineering College

Date: 17th December 2023

Thematic Area: Environment and Energy
Exploring Flexible UV LED for Disinfection of Secondary Treated Municipal Wastewater

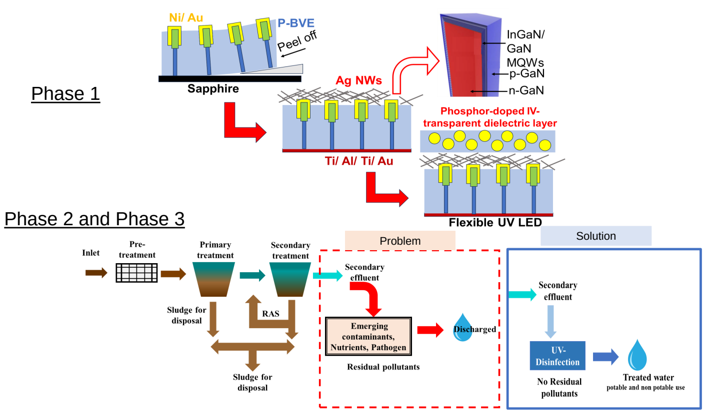
1 Principal investigator and Collaborators

- Principal investigator : Dr. Debajyoti Biswas, Department of Computer Science and Engineering, Shiv Nadar University Chennai
- Collaborator 1 : Dr. Tanusree Sengupta, Department of Chemistry, SSN Engineering College
- Collaborator 2 : Dr. Maria Tchernycheva, CNRS Paris, France
- Collaborator 3 : Dr. Vaishali Choudhary, Shiv Nadar University Chennai
- Student researcher : Hari Kumar, M.Tech. student, Department of Computer Science and Engineering, Shiv Nadar University Chennai

2 Project Summary

Flexible UV LED devices will be investigated as tertiary wastewater treatment unit for microbial deactivation process in this project.The primary objective of the project is to prepare $Lu_3Al_2Ga_3O_2:Er_3^+$ phosphor emitting UVC (ultraviolet-C) coated novel flexible GaN/ InGaN-based LED. The flexible LEDs pumping the phosphors (also known as "LED pumps") are made of self-assembled GaN nanowires (NWs) with radial InGaN/GaN multiple quantum wells (MQWs) in order to produce a ultraviolet emission spectrum. The next phase of the work will try to assess the application of the fabricated UV LED for the disinfection of secondary effluent obtained from sewage treatment plant at Shiv Nadar University Chennai.

3 Graphical Abstract



4 Problem Statement

With dwindling potable water resources, there has been an emphasis on wastewater reuse. Furthermore, many developing and underdeveloped nations suffer from poor sanitation systems which are responsible for accelerated transmission of waterborne disease. Thus, developing an economical and effective water/ wastewater disinfection system is a pressing concern in both the public and private sectors. For many years, the compact design of the UV-disinfection unit and no addition of chemical reagents have served as the upper hand over traditional techniques. Nonetheless, high energy requirements and mercury usage have limited their worldwide application. UV LED technology is energy-efficient and environmentally friendly solution to traditional system for the inactivation of harmful micro-organisms such as bacteria, viruses, and protozoa. Additionally, mechanical flexibility and high stability of the proposed UV-LED system can allow easy integration with large scale filtration system. Accordingly, for the current grant we propose the in-house synthesis of the flexible UV-LED. This will offer the advantages of conformability and integration into larger systems. The developed UV-LED system will be tested for disinfection efficacy and potential of the batch system to damage pathogen and antimicrobial- resistant bacteria present in secondary treated wastewater effluent. Thus, we aim to develop sustainable and scalable UV disinfection-based technologies can ascertain the achievement of UN Sustainable Development Goals 6 and 13.

5 Background Story

Although ultraviolet (UV) disinfection with mercury lamps have been used for many years in the treatment of wastewater and drinking water, traditional mercury-based UV lamps have some detrimental effects on the environment due to its high energy requirements (approximately 400 V), short shelf-life (2,000-10,000 hours) and toxic mercury usage [4],[5]. Hence developing UV LED (Ultra-Violet range Light Emitting Diode) technologies that are climate responsive and scalable, is essential to achieve the confluence of UN Sustainable Development Goals 6 ("Ensure availability and sustainable management of water and sanitation for all.") [1]. The modular nature, scalability, wavelength dependent interactions of UV LED with the capacity of absorbing and blocking contaminants in the wastewater matrix has put UV LED in the forefront of water treatment research. In addition, LEDs are extremely robust devices without a glass covering, and unlike other lighting sources like incandescent, fluorescent, or arc-discharge lamps, their lifespan is not reduced by frequent on/off cycles. The advantage of mechanical flexibility makes the disinfection preventing device more robust, adds a higher degree of freedom, and enhances the compatibility to be integrated in any larger purification system. These advantages of manufacturing cost and space efficiency, durability and customization have led to high usage of flexible LEDs. The process to fabricate UV LED is challenging and to combine the advantages of both flexibility and UV emission, the phosphor coated InGaN/ GaN nanowire blue LED is proposed in this project.

Although flexible organic LED (OLED) performance has improved tremendously over last few decades, they face issues in long term stability, low external quantum efficiency (EQE), low luminance and have limitations in the short wavelength range. Inorganic, mostly nitride-based UV LEDs of different wavelengths in the range of <280 nm (UVC) have been applied for different microorganisms inactivation. In this project, we propose visible to UVC up conversion of Er_{3+} doped Lutetium Aluminium Garnate (LAG) ceramic phosphor. UVC emission characteristics of Er_{3+} is not studied as extensively as its visible range emission characteristics [6],[10] hence this material is explored in this project. Herein lies the novelty of our project that the combination of these specific materials ($Lu_3Al_2Ga_3O_2$:Er^{3+} with InGaN/ GaN nanowires) applied towards wastewater disinfection with the additional advantage of mechanical flexibility is a major challenge to explore. Nucleic acids strongly absorb wavelengths in the expected UVC emission range. Some of the different pathogens and corresponding wavelengths to inactivate them are represented in Figure 1, following [8].

In this context, an additional advantage of mechanical flexibility will be advantageous from the aspects of industrial design and user friendliness. To fabricate a mechanically flexible UV LED is a challenging venture from material selection and device fabrication aspects. To overcome this issue, a combination of phosphor visible to UVC up-converter coating with a nitride-based LED pump is proposed in this project. Er^{3+} doped garnet phosphor materials are already demonstrated in literature [9], we propose to use $Lu_3Al_2Ga_3O_2$:Er^{3+} for this purpose. On other hand, the flexible blue LED pump can be fabricated with InGaN/ GaN multiple quantum well (MQW) nanowires encapsulated in flexible PDMS membrane and peeling off from the saphire substrate , according to the procedures described in [2]. According to the flexibility testing by the authors of [2], there was no discernible degradation when the LEDs were bent down to a maximum of 1.5 cm curvature radius.

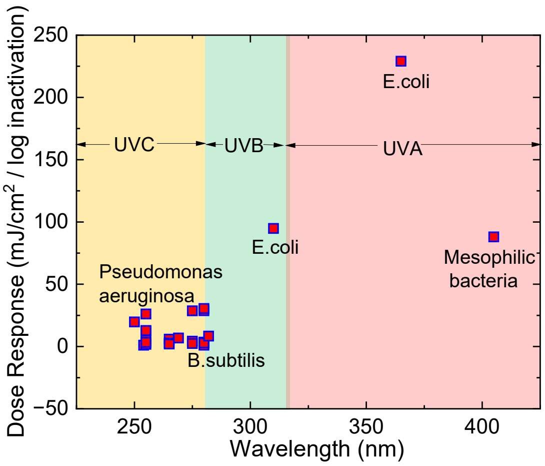


Figure 1: Dose response and wavelength data for water disinfection by UV-LEDs [8]

The future parts of the experiment, will also apply different classification machine learning algorithms, to predict the specific rare earth host material required for inactivation of the particular wastewater contaminant based on the wavelength and dosage per log inactivation data.

6 Approach and Methodology

The project will be carried out in different phases handled by different partners. More detailed descriptions on the methodologies section is discussed in the appendix section.

6.1 Phase 1 : Development of UV LED: PI and Collaborator 2

- **Objective 1: Fabricate GaN/ InGaN nanowires-based flexible blue LED via Metal-organic vapour phase epitaxy (MOVPE).**
- **Objective 2: Dip coat the blue LED with $Lu_3Al_2Ga_3O_2$:Er^{3+} phosphor to convert the blue emission to UV (preferably UVC) emission region.**

Efforts will be carried out to prepare $Lu_3Al_2Ga_3O_2$:Er^{3+} at Shiv Nadar University Chennai and coat it over flexible InGaN/GaN-based blue LED prepared at Dr. Maria Tchernycheva's lab at C2N, France. The Indian team will explore the phosphor material synthesis and learn the flexible blue LED fabrication from the esteemed ' Optoelectronic Devices and INovation (ODIN)' group at C2N.

The experimental method consists of flexible InGaN/GaN nanowire LEDs that function as pumps, doped with a phosphor material. This method, following the procedures reported by Guan et. al.[2] is demonstrated in Figure 2. Further details on LED fabrication is described in appendix 11.1.

6.2 Phase 2: Application of developed UV-LED for secondary treated wastewater : PI, Collaborator 1 and Collaborator 3

- **Water quality analysis:**
  - **Objective 3: Water quality analysis and evaluate the efficacy of the UV LED disinfection process**

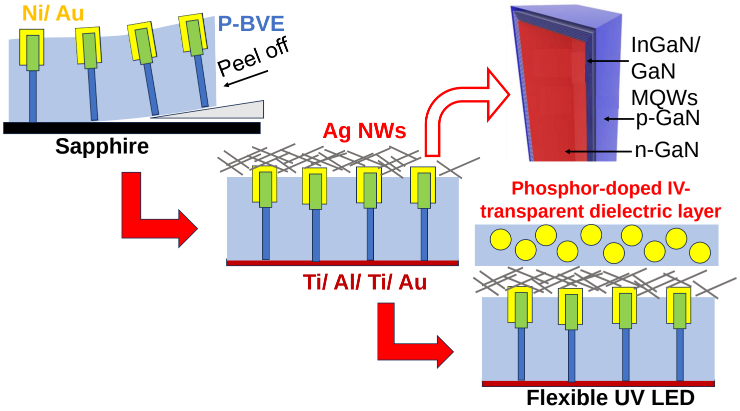


Figure 2: Proposed fabrication method for flexible InGaN/ GaN based UV LED

Municipal secondary treated wastewater will be collected from the Sewage treatment plant located at SNU Chennai. The wastewater samples will be collected and analyzed for water quality parameters as per APHA 2017. To check disinfection efficacy, batch reactor will consisting of UV LED lamp will be connected to DC power supply and will be operated with a constant forward current mode. The batch reactor will contain an array of UV-LEDs. The array will be fixed facedown above the surface of a sample. The samples will be magnetically stirred throughout the experiment to reduce mass transfer resistance. The wastewater sample will be collected at regular intervals as tested for microbial activity.

– **Objective 4: Investigating antimicrobial activity**

To investigate the antimicrobial effect of the developed UV-LED, both broth dilution and spread plate method ([3],[7]) will be followed. The effect will be assessed against two-three different strains of both gram positive and gram-negative bacteria as pathogenic representatives as described in appendix 11.2.

– **Objective 5: Stability and Fouling test of the UV-LED:**

For practical application purposes, the stability and fouling study will be carried out. The UV-LED system will be run continuously for an extended period and UV output will be measured. Impact of varying operating conditions, including temperature and relative humidity, will be measured. The UV-LED devices will undergo repeated on-off cycles to simulate real-world usage patterns and evaluate the stability of UV output.

7 Expected Outcome

The aforementioned research collaboration holds immense promise for advancing the field of wastewater disinfection through the fabrication of ultraviolet C (UVC) range GaN/InGaN light-emitting diodes (LEDs). This project is anticipated to yield significant outcomes, addressing some of the pressing challenges associated with wastewater treatment.

– **Environmental Benefits:** Disinfection of wastewater is a critical concern, given the escalating global water pollution crisis. Conventional treatment methods are often insufficient in effectively eliminating persistent organic pollutants, microorganisms, and emerging contaminants from wastewater. The utilization of UVC light, with its shorter wavelength, has shown great potential for disinfection and the breakdown of harmful contaminants.

– **Novelty in Device Fabrication Technology:** GaN/InGaN LEDs operating in the UVC range are at the forefront of this innovation, and the research collaboration is targetting to make several significant contributions in the area of device flexibility, applications of novel LED fabrication technology and corresponding application area.

- **Collaborative Networks leading to high quality publication :** The collaborative research between institutions in India and France will promote knowledge exchange and cross-cultural learning. It will also strengthen international research networks, fostering a global approach to solving pressing water quality issues. We plan to write two research papers based on this collaborations: (a) on the flexible LED fabrication aspect of the project and (b) on the application of GaN/InGaN LED for secondary effluent disinfection..
- **Commercial Viability:** With successful outcomes, this research may pave the way for the commercialization of advanced UVC LEDs for wastewater treatment. This can create economic opportunities for both countries and contribute to the global water industry.

In conclusion, the research collaboration between Dr. Debajyoti Biswas from Shiv Nadar University, Chennai, Dr. Tanusree Sengupta from SSN Engineering College, Dr. Vaishali Choudhary from Shiv Nadar University Chennai and Dr. Maria Tchernycheva from CNRS Paris holds great potential for significantly advancing the field of water disinfection through the development of UVC range GaN/InGaN LEDs. The anticipated outcomes encompass improvements in efficiency, materials, reliability, and scalability, with the ultimate goal of providing sustainable, cost-effective, and environmentally friendly solutions for addressing the pressing challenges of water pollution and wastewater treatment on a global scale. This collaborative effort also exemplifies the power of international cooperation in solving critical environmental issues.

8 Fund Utilization Plan

BUDGET FOR MINOR EQUIPMENT

Serial No.	Generic name of the Equipment along with make and model	Imported/ Indigenous	Estimated Costs	Spare time for other users (in %)
A	Hallmarc Spin Coater with UV Curing	Indigeneous (Holmarc)	INR 2,55,240	30
B	HACH Water Quality Kit	Imported	INR 2,50,000	50
	Total		INR 5,05,240	

BUDGET FOR CONSUMABLES

Serial No.	Item	1st Year Budget	2nd Year Budget	3rd Year Budget	Total (in INR)
A	Consumables costs (LED Fabrication materials, wastewater disinfection characterization materials, laboratory space set up costs, furnitures etc)	6,00,000	5,50,000	4,50,000	16,00,000

BUDGET FOR TRAVEL AND ACCOMMODATION

Serial No.	Item	1st Year Budget	2nd Year Budget	3rd Year Budget	Total (in INR)
A	Domestic travel (To CFTIs for fabrication and characterization)	16,000	16,000	16,000	48,000
B	International Travel (To collaborator's institute and return)	150,000	150,000	150,000	450,000
C	International Accommodation (In Paris)	2200 Euro	2200 Euro	2200 Euro	6600 Euro or approx. INR 590,205
	Total				INR 10,88,205

BUDGET FOR MANPOWER

Serial No.	Item	1st Year Budget	2nd Year Budget	3rd Year Budget	Total (in INR)
A	JRF salary	150,000	150,000	150,000	450,000

BUDGET FOR OTHER COSTS/CONTINGENCIES

Serial No.	Item	1st Year Budget	2nd Year Budget	3rd Year Budget	Total (in INR)
A	Other costs/Contingency costs	10,000	10,000	10,000	30,000

TOTAL BUDGET

Serial No.	Area of Funding	Estimated Costs
A	Budget For Minor Equipment	INR 5,05,240
B	Budget for Consumables	INR 16,00,000
C	Budget For Travel And Accomodation	INR 10,88,205
D	Budget For Manpower	INR 4,50,000
E	Budget For Contingencies	INR 30,000
	Total	INR 3,673,445

9 Collaborating Institution's Role

Collaborative research is at the heart of scientific progress, bringing together experts from different fields to tackle complex challenges. A prime example of such collaboration is the ongoing research between Dr. Debajyoti Biswas from Shiv Nadar University, Dr. Vaishali Chowdhary from Shiv Nadar University Chennai, Dr. Tanusree Sengupta from SSN Engineering College and Prof. Maria Tchernycheva from Centre For Nanoscience and Nanotechnology, CNRS Paris in the quest to fabricate flexible UV light-emitting diodes (LEDs) for secondary treated municipal wastewater disinfection. This part of the proposal delves into the crucial roles each collaborator plays in this innovative experiment.

Dr. Debajyoti Biswas:
The PI Dr. Debajyoti Biswas is a key player in this collaboration, with his expertise spanning conducting and semiconducting polymers, conformable electronic devices, microelectronics, and the application of device physics in electronics. His role is multifaceted, encompassing the design and optimization of flexible UV LEDs targetted towards wastewater disinfection.

Dr. Tanusree Sengupta:
Dr. Tanusree Sengupta's background in protein biochemistry adds a unique dimension to the bacteria deactivation project. Her expertise in characterizing UV wastewater disinfection is crucial in assessing the effectiveness of the developed flexible UV LEDs.

Prof. Maria Tchernycheva:
Prof. Maria Tchernycheva, hailing from CNRS Paris, is a world-renowned expert in the fabrication and testing of optoelectronic devices, particularly those based on semiconductor nanowires. Her research focus on nanowire flexible light-emitting diodes, solar cells, and piezosensors is directly aligned with the goals of the experiment. Prof. Tchernycheva brings her wealth of experience and expertise to the table, guiding the team in the intricate process of fabricating InGaN/GaN-based UV LEDs. Her extensive publication record reflects her dedication

to advancing the field.

Dr. Vaishali Chowdhary:
Another important collaborator in this endeavor is Dr. Vaishali Chowdhary, who specializes in water and wastewater treatment. Vaishali's experience and knowledge complement Dr. Sengupta's role in characterizing the municipal wastewater disinfection efficiency. As an expert in this specific application, Vaishali offers insights and expertise that are invaluable in optimizing the UV LEDs for wastewater treatment. By working alongside engineers and materials scientists, Dr. Chowdhary ensures that the fabricated UV LEDs meet the necessary criteria for wastewater treatment, bridging the gap between materials science and environmental science.

Serial No.	Name	E-mail ID	Phone No.	Role
A	Dr. Debajyoti Biswas	debajyotib@snuchennai.edu.in	9176338192	PI
B	Dr. Tanusree Sengupta	tanusrees@ssn.edu.in	99404 01250	Collaborator 1
C	Dr. Maria Tchernycheva	maria.tchernycheva@c2n.upsaclay.fr	+33 1 69 15 40 51	Collaborator 2
D	Dr. Vaishali Choudhary	vaishalic@snuchennai.edu.in	9560947825	Collaborator 3

10 Timelines

	Year 1 Semester 1	Year 1 Semester 2	Year 2 Semester 1	Year 2 Semester 2	Year 3 Semester 1	Year 3 Semester 2
Equipment and materials purchase	■					
LED fabrication and characterization process		■	■			
UV-emissive phosphor layer fabrication and characterization				■	■	
Experiments on wastewater disinfection					■	■

11 Further Investigation Aspects

11.1 Application of Machine Learning

Machine learning algorithms can be applied to predict the specific rare earth host material required for inactivation of the particular wastewater contaminant based on the wavelength and dosage per log inactivation.

References

[1] Jamie Bartram et al. "Policy review of the means of implementation targets and indicators for the sustainable development goal for water and sanitation". In: *npj Clean Water* 1.1 (Apr. 2018), p. 3. ISSN: 2059-7037. DOI: 10.1038/s41545-018-0003-0. URL: https://doi.org/10.1038/s41545-018-0003-0.

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12 Appendix

The following appendix sections describe the experimental methods in a more detailed manner.

12.1 Phase 1 : Development of UV LED: PI and Collaborator 2

In this phase, the flexible LEDs pumping the phosphors (also known as "LED pumps") are made of self-assembled GaN nanowires (NWs) with radial InGaN/GaN multiple quantum wells (MQWs) in order to produce a ultraviolet emission spectrum. Metal-organic vapour phase epitaxy (MOVPE) is used to build the NW arrays on sapphire substrates. For producing a satisfactory ohmic contact, the NW base sections can first be coated with a resist layer. Next, a thin layer of Ni/ Au can be formed on the p-GaN shells of NWs and annealed at 400°C in an oxygen-containing environment. After this, the phosphor powder $Lu_3Al_2Ga_3O_2$:Er^{3+} can be combined with poly(benzyl vinyl ether)(p-BVE) at a mass ratio of 1:10 to create few hundred μm-thick p-BVE membranes, which will be used to manufacture the flexible UV LED. To change the emission from the LED pump into the UV emission spectrum, these membranes can be placed on the top surface of the LED pump layer.

To convert the blue InGaN/ GaN LED to UV LED, oxides of all prerequisite materials, lutetium oxide (Lu_2O_3), aluminium oxide (Al_2O_3) and erbium oxide (Er_2O_3), can be used as precursors (weighed and combined in a stoichiometric ratio) for the synthesis of the proposed phosphor material using high temperature solid state reaction, following the procedures described by Tamrakar et. al.[9]. H_3BO_3 can be employed as a flux, in modest amounts. After being moved to an agate pestle and mortar, the mixture can be ground for one hour to mix the reagents. This resultant mixture will be transferred to an alumina crucible, and heated to 1550°C for four hours in a muffle furnace and naturally cooled to 25°C. The blue LEDs can be dipped into phosphor sol solution, followed by drying for 1 h and annealing at 104°C.

12.2 Phase 2 : Application of developed UV-LED for wastewater disinfection : PI, Collaborator 1 and Collaborator 3

Cell culture condition: Luria Burtani (LB) medium can be utilized as a liquid medium containing tryptone, yeast extract and NaCl (10 g) in 1,000 mL sterile distilled water at pH 7.0. Besides, LB agar containing tryptone, yeast extract, NaCl, and agar powder in 1,000 mL of sterile distilled water of at 7.0 was used as a solid medium for spread plate study. For all the bacteria, a representative single colony can be picked up with a wire loop and that loopful of culture can be spread on LB agar slant to give single colonies and incubated at 37°C for 24 h. These fresh overnight cultures of all the bacteria can further be diluted as required to give a working concentration in the range of 106–109 colony forming units (cfu/mL) before every experiment.

Antibacterial assay: Bacterial culture of every strain at the desired working concentration will be aliquoted in different test tubes and each one will be subjected to different power of UV-LED for different time period. Subsequently, they will be grown for 24 h at 37°C in a shaker incubator. The optical density of all the solutions will be measured before and after incubation at 650 nm. Bacterial culture without exposure to UV-LED will be used as a control. For the spread plate experiments, the test tubes containing desired concentration of bacterial culture will be subjected to UV exposure as described before. Cultures will then be inoculated at 37°C for 5–7 h. Then, 50 mL from each tube will be spread onto LB agar plates inside laminar flow. Finally, plates will be incubated at 37°C for 24 h, and the viable cells will be counted and compared with a control plate.