

## Indian Science Technology and Engineering facilities Map (I-STEM)

### **Partial financial support for using R&D facilities in other institutions through the I-STEM web portal to Users from Academic Institutions and Start-ups (Format-1)**

*Format for requesting support for accessing and using facilities through the I-STEM web portal.*

#### **(i) Details of the Personal Information**

- a. Name of the Researcher/Faculty Member: Gaius Cherian Mathew
- b. Name of the Institution/Start-up: National Institute of Technology Calicut
- c. Name of the Department: School of Materials Science and Engineering
- d. Degree registered for/Product being developed (for start-up): Ph.D
- e. Mob. No. 9037760826
- f. Email ID: gaiuscherian@gmail.com, gaius\_p210091mt@nitc.ac.in

#### **(ii) Project/Research topic Details:**

- a. Project Title/Research Title: Titanium-based nanostructured High Entropy Materials as Photocatalyst for Solar Fuel Synthesis
- b. Sub area: Photocatalytic CO<sub>2</sub> Reduction
- c. Area Description:

Methods to convert CO<sub>2</sub> to valuable and useful products can not only change the current global warming scenario, but also net-zero emission could be ensured. Since CO<sub>2</sub> has high thermodynamic stability, the photocatalytic reduction process is efficient and economical. High entropy materials (HEMs) exhibit high stability, good structural and functional properties, and remarkable photocatalytic reduction ability. HEMs in microscale constituting transition metals with d<sup>0</sup> configuration display multifold photoreduction capability when compared to widely researched TiO<sub>2</sub>. Photocatalytic activity of nanoscale-HEMs has not yet been investigated, and its increased surface area could exponentially enhance the performance.

- d. Aims & Objective (in Bullets):

- Obj. 1: Synthesis of Ti-based HEO nanoparticles using facile wet chemistry methods.
- Obj. 2: Characterization of synthesized HEOs and their structure-based comparative study.

- e. Duration (MM/YYYY): 10/2022 to 03/2023; within 6 months

- f. Project Summary:

The long-term CO<sub>2</sub> emissions have increased from 6 Gt CO<sub>2</sub>/yr in 1900 to 43 Gt CO<sub>2</sub>/yr in 2019, as per the sixth assessment report of the IPCC (Intergovernmental Panel on Climate Change) 2022. Reducing CO<sub>2</sub> to other valuable products, thus ensuring a net-zero emission, is considered the panacea for this burning issue. Photocatalytic reduction is a green method, and efficient tailoring of catalyst could develop value-added fuels from CO<sub>2</sub>. The three main factors of photocatalytic reduction are:

- (1) the formation of charge carriers,

- (2) charge separation and transport to the surface, and
- (3) adsorption and reduction of  $\text{CO}_2$  [1].

These three processes are depicted in Fig. 1. Modification techniques to improve any of the above three factors could result in the overall enhancement of the photocatalytic activity. Recombination of charge carriers in the bulk or at the surface of the photocatalyst is an undesirable process, which reduces the overall efficiency of the photocatalyst. As the charge carriers formed increases, more and more  $\text{CO}_2$  could be adsorbed and activated. This could be enhanced by increasing the surface area of the photocatalysts, designing narrow bandgap photocatalysts, addition of dye sensitizers and by quantum confinement effects. Efficient charge separation can be ensured either by the formation of a heterojunction photocatalyst or by the addition of noble metal nanoparticles to the photocatalysts, which could act as an electron trap.  $\text{CO}_2$  adsorption can be enhanced by exposing more adsorption sites without compromising on the gas diffusion adsorption kinetics [2].

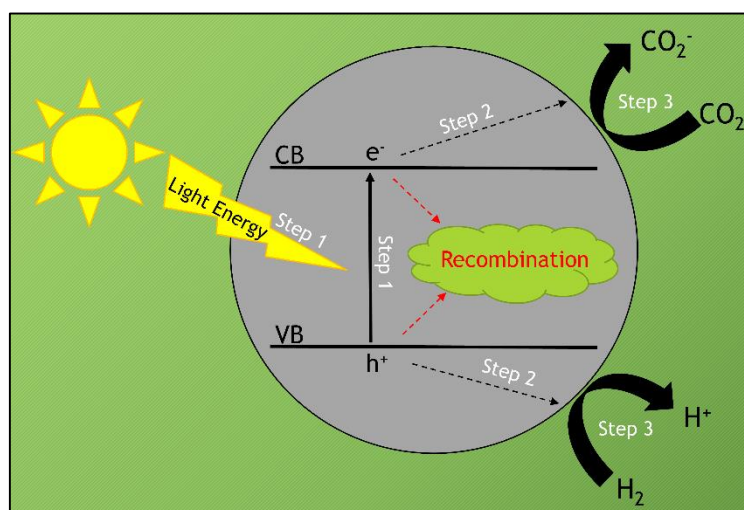


*Fig. 1. Processes involved in photocatalytic  $\text{CO}_2$  reduction.*

Semiconductor-based systems, especially  $\text{TiO}_2$  and its modifications, are the most investigated since it acts as both the catalyst and sensitizer, is non-toxic and, stable in nature [3–5]. But the reduction efficiency and product selectivity is low, when compared to other biological or metal complex-based systems. Biological systems have high selectivity but very low efficiency and, metal-complex systems have very low selectivity. Semiconductor systems are not complex in nature and further modifications can greatly enhance the photocatalyst properties [6,7].

High Entropy Materials (HEMs) are a class of materials constituting of 5 or more principal elements and each with an atomic percentage ranging between 5-35% [8]. High Entropy Alloys (HEAs) and Ceramics (HECs) are classes of HEM, which shows catalytic activity for  $\text{CO}_2$  reduction [9]. HECs consists of High Entropy Oxides (HEOs) and Nitrides (HENs) which exhibit excellent photocatalytic activity. Transition metals like Ti with  $d^0$  configuration are proven to be photocatalytically active. HEOs and HENs constituting transition metals are promising photocatalysts for the future, since

they are highly stable and multifold product yield is displayed when compared to  $\text{TiO}_2$  [10–12]. But the current study in photocatalytic HEOs are limited and researchers have studied HEOs only upto microscale dimension [13]. Exposing HEOs to the nanoregime can exponentially increase the photocatalytic activity, as the increased surface area leads to more  $\text{CO}_2$  adsorption and activation sites. The quantum confinement effects could also increase the light harvesting properties.

The initial part of the project mainly aims to synthesize selected HEO as per the literature. HEO photocatalyst constituting of Ti, Zr, Nb, Hf and Ta, is studied in the micro dimensions and enhancement in performance has to be studied when the size is further reduced. Nanoparticles can be prepared using a fast microwave-assisted irradiation method by Kheradmandfard et al [16]. The synthesized material have to be characterized using several techniques like XRD, SEM-EDS, HR-TEM, XPS, BET, UV-Vis-NIR spectroscopy and photoluminescence study. These characterization tests conducted will help to understand the morphology and size of the system, to ensure the bonding between the constituent principal elements and to determine the crystal structure of synthesized HEMs.

The present project focuses on the synthesis of nano-dimensional HEO and its characterization. Selected HEO constituting (Ti, Zr, Nb, Hf, Ta, O) will be synthesized using wet chemistry methods. It is proposed that highly stable and photocatalytically active nanostructured HEM can be developed by this study.

g. **Technical Details**

a. **Technology Readiness Level (TRL):**

The scope of this project include the synthesis and characterization of nano-dimensional high entropy materials for photocatalytic  $\text{CO}_2$  reduction. The size, structure and morphology of synthesized HEO are analyzed to study its photocatalytic ability. Hence, the Technology Readiness Level (TRL) of this project is 3.

b. **Methodology:**

The proposed project is to be executed in three stages and the methodology is as follows:

**Stage I : Synthesis of HEM nanoparticles:**

- Synthesis of nanoparticles by a fast microwave-assisted irradiation method, as mentioned by Kheradmandfard et al. [16].

**Stage II: Characterization of HEM nanoparticles:**

- The characterization techniques comprised in the second stage of the study are:

1. X-Ray Diffraction (XRD),

2. Scanning Electron Microscopy (SEM) with Energy dispersive spectroscopy (EDS)-Elemental Mapping,
3. High-Resolution Transmission Electron Microscope (HR-TEM),
4. X-Ray Photoelectron Spectroscopy (XPS),
5. UV-Vis-NIR Spectroscopy with Diffuse Reflectance Spectroscopy (DRS),
6. Photoluminescence emission spectroscopy (PL) and
7. Brunauer-Emmett-Teller (BET) analysis
8. Reduction potential analysis by Linear Sweep Voltametry (LSV).

c. Design & development:

Despite the efforts of numerous researchers to develop a highly efficient HEM-based photocatalyst, the research is still in the nascent stage. HEO consisting of Ti, Zr, Nb, Hf, Ta and O is selected for synthesis and its structure and morphology will be studied. The selected HEO will be synthesized using facile methods to develop corresponding nanoparticles. Characterization of synthesized HEM will be carried out to understand the morphology, structure and to confirm the formation of HEM. The comparative study on structure and morphology of HEO will help to correlate it to the photocatalytic performances of these HEMs, which is to be studied in the future.

The proposed project is to be executed in two stages. The first stage comprises of the synthesis of HEO as nanoparticles through facile methods. The second stage involves the characterization of synthesized HEO. A comparative study of the structure of synthesized HEMs shall also be done in this stage. The detailed experimental approach followed in each phase is described as follows:

**Stage I : Synthesis of HEO nanoparticles:** Stage I consists of the synthesis of HEO as nanoparticles, through facile method mentioned in the literature. Nanoparticles can be prepared using a fast microwave-assisted irradiation method, as mentioned by Kheradmandfard et al. [16]. Equimolar amounts of principal element precursors are separately dissolved in deionized water. It is then sequentially added to an ammonium hydroxide solution ( $\text{NH}_4\text{OH}$ , pH 12). Microwave irradiation at 850 W and 2.45 GHz for 3 min in the ambient air will synthesize the HEO nanoparticles as a precipitated powder separated using centrifugation. The separated particles are then washed using ethanol followed by deionized water, and then dried at 80 °C for 1 h in an oven. Fig. 2 shows the HEO nanoparticle-synthesis route.

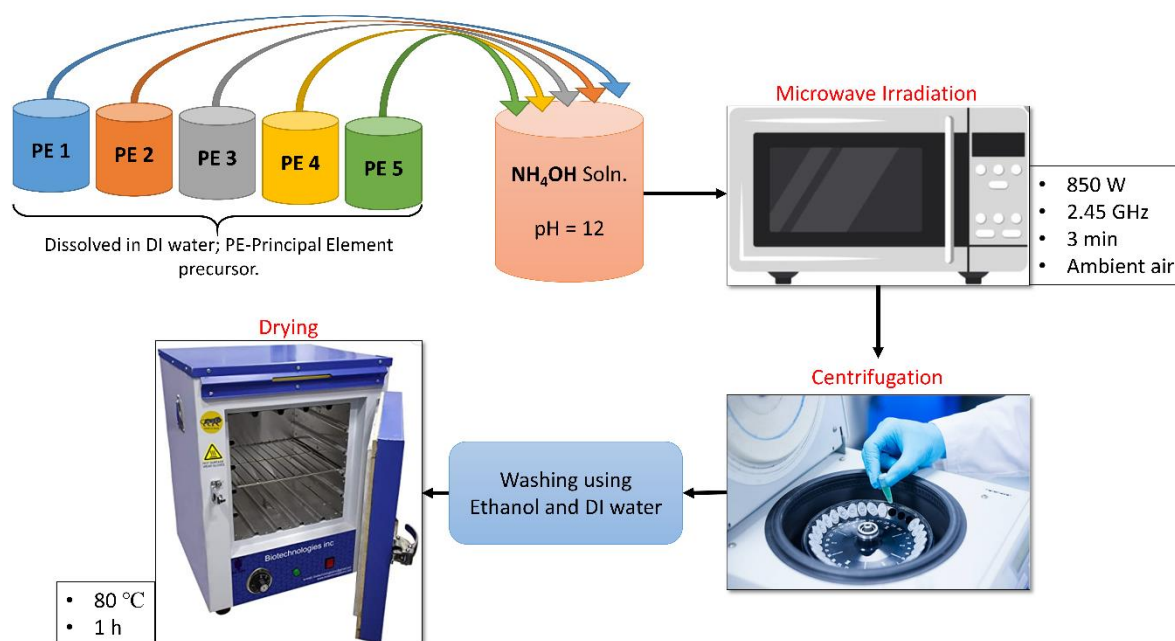


Fig. 2. Steps involved in HEM nanoparticle synthesis.

**Stage II:** In the last stage, the characterization of synthesized samples will be conducted. HEO Nanoparticles will be characterized to confirm the formation of high entropy material and also to study the morphological properties and atomic bonding. The characterization techniques required are listed below:

1. **X-Ray Diffraction (XRD):** To evaluate the presence of all principal elements within the HEOs and study the crystal structure of HEO.
2. **Scanning Electron Microscopy (SEM) & High-Resolution Transmission Electron Microscope (HR-TEM):** To study the morphology of the photocatalyst and particle size.
3. **Energy dispersive spectroscopy (EDS)-Elemental Mapping:** To quantify the atomic weight percentages of principal elements in HEO.
4. **X-Ray Photoelectron Spectroscopy (XPS):** To find the oxidation states of different principal elements present.
5. **UV-Vis-NIR Spectroscopy with Diffuse Reflectance Spectroscopy (DRS):** To study the light absorption characteristic of HEO and calculate its bandgap.
6. **Photoluminescence emission spectroscopy (PL):** To study the lifetime of excited electrons and thereby understand the charge separation enhancement.
7. **Brunauer-Emmett-Teller (BET) analysis:** To calculate the specific surface area of HEO.
8. **Linear Sweep Voltammetry (LSV):** To study the reduction potential of synthesized photocatalyst material.
9. Detection of photocatalytic activity using methylene blue degradation method.

d. Facilities and Instrumentation required:

The successful completion of the project depends on the proper synthesis, characterization and the availability of facilities. For the synthesis of HEM nanoparticles, centrifuge and hot air oven are available at the institute.

For the characterization stage, the facilities which are available within the institute and outside are mentioned in Table.

*Table 1. Availability of characterization facilities required.*

| Sl.No | Facility Required | Availability within the institute | I-STEM Availability           |
|-------|-------------------|-----------------------------------|-------------------------------|
| 1.    | XRD               | Yes                               | -                             |
| 2.    | SEM-EDS           | Yes                               | -                             |
| 3.    | HR-TEM            | No                                | STIC-CUSAT                    |
| 4.    | XPS               | No                                | Alagappa University           |
| 5.    | UV-Vis-NIR        | No                                | STIC-CUSAT                    |
| 6.    | PL                | No                                | Christian College, Chengannur |
| 7.    | BET               | Yes                               | -                             |
| 8.    | LSV               | Yes                               | -                             |

e. Consumables required:

The reference material selected is Degussa P25 TiO<sub>2</sub> nanopowder. Powder oxides of proposed transition metals like Ti, Nb, Zr, Hf and Ta have to be purchased for making the metal precursor solution. NH<sub>4</sub>OH solution is a necessary reagent for both the synthesis methods. Ethanol and DI water are available at the department laboratory.

*Table 2. List of chemicals and consumables required.*

| Category                | Product                               |
|-------------------------|---------------------------------------|
| Chemicals               | TiO <sub>2</sub> powder               |
|                         | NbO <sub>2</sub> powder               |
|                         | ZrO <sub>2</sub> powder               |
|                         | HfO <sub>2</sub> powder               |
|                         | Ta <sub>2</sub> O <sub>5</sub> powder |
|                         | Ammonia solution                      |
|                         | Methylene Blue                        |
|                         | Ethanol                               |
| Glass and plastic wares | Centrifuge tubes                      |
|                         | 100 ml glass beakers                  |
|                         | 50 ml glass beakers                   |

h. Expected output and outcome:

The proposed work is expected to successfully synthesize and characterize High Entropy Oxide nanoparticles. Such HEM can possibly serve as highly stable and efficient photocatalysts for CO<sub>2</sub> reduction. The characterization techniques mentioned also defines and completely describes the developed HEM. The novel work proposed in the project can be published in SCI-indexed journals and also can be used as a reference for future photocatalyst-based research.

- i. Industry/Institute Collaboration: NO
- (i) Name of the Collaborating Industry/Institute
- (ii) Collaboration Details
- (iii) Additional Details if any

j. **Funding required from I-STEM:**

1) **Chemicals and Consumables:**

The funding required is mostly for the chemicals and consumables part of the project. For the synthesis of transition metal based-High entropy materials, as per the literature, oxides of transition metals are used. Precursor powders of Ti, Zr, Nb, Hf and Ta are currently proposed to purchase in low volumes of 20 – 25 g. Majority of the funding goes towards the procurement of these base materials. Other solutions and reagents such as ammonia solution (NH<sub>4</sub>OH), ethanol and DI water are also required for nano-HEO synthesis. DI water is available at the department laboratory. The list of chemicals and reagents along with their quantities and price are shown in Table 1. The quantity of transition metal oxide mentioned in the table is the lowest provided by the supplier. The price of transition metal oxide powders and methylene blue are quoted from Sigma-Aldrich, India and that of ammonia solution from SRL Chemicals, India.

*Table 3. Details of consumables required for this project.*

| Sl. No.                | Chemicals/Consumables                     | Brand         | Quantity/Capacity | Price (₹)       |
|------------------------|-------------------------------------------|---------------|-------------------|-----------------|
| 1.                     | TiO <sub>2</sub> nanopowder               | Sigma-Aldrich | 25 g              | 5580.00         |
| 2.                     | Nb <sub>2</sub> O <sub>5</sub> nanopowder | Sigma-Aldrich | 25 g              | 3013.68         |
| 3.                     | ZrO <sub>2</sub> nanopowder               | Sigma-Aldrich | 25 g              | 18186.00        |
| 4.                     | HfO <sub>2</sub> nanopowder               | Sigma-Aldrich | 25 g              | 7274.40         |
| 5.                     | Ta <sub>2</sub> O <sub>5</sub> nanopowder | Sigma-Aldrich | 10 g * 2          | 26837.40        |
| 6.                     | Ammonia solution                          | SRL Chemicals | 500 ml            | 300.00          |
| 7.                     | Methylene Blue                            | Sigma-Aldrich | 25 g              | 4416.00         |
| 8.                     | Ethanol                                   | Lab Grade     | 500 ml            | 600.00          |
| 9.                     | Centrifuge tubes                          | Tarsons       | 15 ml * 50        | 1000.00         |
| 10.                    | 100 ml glass beaker                       | Borosil       | 3 Nos.            | 500.00          |
| 11.                    | 50 ml glass beaker                        | Borosil       | 10 Nos.           | 1000.00         |
| <b>Total estimated</b> |                                           |               |                   | <b>68707.43</b> |

2) **Equipments required:**

A microwave oven (850 W, 2.45 GHz) for the synthesis of nano-HEOs is also an essential piece of equipment required. Microwave irradiation methods are considered to be an efficient and fast synthesis route. It ensures a simple and facile synthesis of HEO nanoparticles. The expected price of microwave oven with required specification is mentioned in the table below.

*Table 4. Details of equipment required for this project*

| Sl.No                  | Equipment      | Make/Model | Specification   | Price (₹)       |
|------------------------|----------------|------------|-----------------|-----------------|
| 1.                     | Microwave Oven | IFB, 25BC4 | 900 W, 2.45 MHz | 13000.00        |
| <b>Total estimated</b> |                |            |                 | <b>13000.00</b> |

### 3) **Characterization:**

The characterization part of this project is very important as it confirms the synthesis of nanostructured HEOs. The studied samples are: synthesized HEO and the reference material, Degussa P25 TiO<sub>2</sub>. As mentioned earlier, some of the facilities are available at NIT Calicut itself and for availing some facilities, outside institutions must be approached. UV-Visible-NIR spectroscopy and HR-TEM will be conducted from the Sophisticated test and instrumentation centre (STIC) under Cochin University of Science and Technology (CUSAT) Ernakulam, Kerala. STIC-CUSAT has been chosen as it is among the nearest I-STEM institute from NIT Calicut (around 180 Kms). Furthermore, STIC-CUSAT offers relatively lower testing charges for the characterization techniques mentioned. Photoluminescence (PL) study will be conducted at PSG Institute of Advanced Studies (IAS), Coimbatore, Tamil Nadu. XPS study will be done at Alagappa University, Karaikudi, Tamil Nadu. SEM-EDS analysis, XRD, BET and LSV analysis are available at NIT Calicut itself. The total estimate for characterization is shown in Table 2.

*Table 5. Details of characterization facilities required for the study.*

| Sl. No | Characterization | Availability                                    | Rate per Sample (INR) | Total Rate per Instrument (₹) |
|--------|------------------|-------------------------------------------------|-----------------------|-------------------------------|
| 1.     | HR-TEM           | STIC-CUSAT                                      | 2301.00               | 9204.00                       |
| 2.     | UV-Vis-NIR       | STIC-CUSAT                                      | 378.00                | 1512.00                       |
| 3.     | PL               | Christian College, Chengannur                   | 590.00                | 2360.00                       |
| 4.     | XPS              | Alagappa University (ALU), Karaikudi, Tamilnadu | 1500.00               | 6000.00                       |
| 5.     | XRD              | NIT Calicut                                     | 200                   | 800.00                        |
| 6.     | SEM-EDS          | NIT Calicut                                     | 500                   | 2000.00                       |

|                        |     |             |     |                 |
|------------------------|-----|-------------|-----|-----------------|
| 7.                     | BET | NIT Calicut | 600 | 2400.00         |
| 8.                     | LSV | NIT Calicut | 200 | 800.00          |
| <b>Total estimated</b> |     |             |     | <b>24276.00</b> |

### **References:**

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- [3] W. Hou, W.H. Hung, P. Pavaskar, A. Goepfert, M. Aykol, S.B. Cronin, *Photocatalytic conversion of CO<sub>2</sub> to hydrocarbon fuels via plasmon-enhanced absorption and metallic interband transitions*, *ACS Catal.* 1 (2011) 929–936. <https://doi.org/10.1021/cs2001434>.
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**Details of Requirements & Mode of the Engagement:** (Attach the quotation for the consumables)

(i) Short term Engagement:

a. Usage of testing/characterization facilities across the country (Characterization Mode)

i. Testing Charges

*Table 6. Characterization facilities required and an estimate for the same.*

| Sl. No                 | Instrument/Facility | Availability      | Testing charge per Sample (INR) | Total charge per Instrument (₹) |
|------------------------|---------------------|-------------------|---------------------------------|---------------------------------|
| 1.                     | HR-TEM              | STIC-CUSAT        | 2301.00                         | 9204.00                         |
| 2.                     | UV-Vis-NIR          | STIC-CUSAT        | 378.00                          | 1512.00                         |
| 3.                     | PL                  | Christian College | 590.00                          | 2360.00                         |
| 4.                     | XPS                 | ALU               | 1500.00                         | 6000.00                         |
| 5.                     | XRD                 | NIT Calicut       | 200.00                          | 800.00                          |
| 6.                     | SEM-EDS             | NIT Calicut       | 500.00                          | 2000.00                         |
| 7.                     | BET                 | NIT Calicut       | 600.00                          | 2400.00                         |
| 8.                     | LSV                 | NIT Calicut       | 200.00                          | 800.00                          |
| <b>Total estimated</b> |                     |                   |                                 | <b>24276.00</b>                 |

ii. Consumable charges

*Table 7. Total estimated for chemicals and consumables,*

| Sl. No                 | Consumables               | Estimated charges (₹) |
|------------------------|---------------------------|-----------------------|
| 1.                     | Chemicals and consumables | 68707.43              |
| <b>Total estimated</b> |                           | <b>68707.43</b>       |

iii. Travel Cost (Boarding and Lodging, if any)

The travel costs vary depending on the testing centre. At least four times up and down visit is required for completing the project.

*Table 8. Total estimated for travel and boarding expenses.*

| Sl. No                 | I-STEM Centre       | Estimated charges (up and down) (INR) | Boarding and lodging per day (₹) |
|------------------------|---------------------|---------------------------------------|----------------------------------|
| 1.                     | STIC-CUSAT          | 400.00                                | Not required                     |
| 2.                     | PSG-IAS, Coimbatore | 400.00                                | 600.00                           |
| <b>Total estimated</b> |                     |                                       | <b>7200.00</b>                   |

**The total expense is estimated to be:**

*Table 9. Total estimate for the project.*

| Sl. No.               | Category           | Amount (₹)       |
|-----------------------|--------------------|------------------|
| 1.                    | Testing charges    | 24276.00         |
| 2.                    | Equipments         | 13000.00         |
| 3.                    | Consumables        | 68707.43         |
| 4.                    | Travel and lodging | 7200.00          |
| <b>Total expenses</b> |                    | <b>108167.43</b> |

***(Round-trip to the Host Institution by 2<sup>nd</sup> class (Non-AC) railway via the nearest route)***

Details of User's Bank Account:

**Beneficiary Name (User's name):** Mr. Gaius Cherian Mathew      **PAN:** FGLPM4274C

**Bank Account No:** 38597395360

**Name of the Branch:** Kattodu

**IFSC Code:** SBIN0070437

***Note: Proposed proposal must be executed within stipulated duration (max. 6 month) to avoid fund availability issues. Travel form will be provided by the I-STEM office. The users will submit the technical report, original invoice (duly signed by the Head of the Institution) raised by the Host Institution through I-STEM portal only along with a copy of the travel forms and original tickets through post to the Nodal Officer, I-STEM, Indian Institute of Science, Bangalore. A scan copy of all the documents may be sent in advance.***

***Declaration:***

We have read the terms and conditions for the I-STEM scheme under “*Partial financial support for using R&D facilities through the I-STEM web portal to Users from Academic Institutions and Start-ups (Proposal Format-I)*”

a) Researcher/ PhD scholar: Mr. Gaius Cherian Mathew



b) Faculty member/ Guide: Dr. Shijo Thomas



30-09-2022  
NIT Calicut