

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

Invites application for

1. **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:	AISWARYA V
b. Name of the Institution:	NATIONAL INSTITUTE OF TECHNOLOGY CALICUT, KERALA
c. Name of the Department:	DEPARTMENT OF CHEMICAL ENGINEERING
d. Degree registered for:	PhD
e. Mob. No.	9747615310; 9633675310
f. Email ID:	aiswarya_p190130ch@nitc.ac.in

(ii) Project/Research topic Details:

a. Project Title/Research Title:	Development of a novel composite for thermal energy storage by incorporating the microencapsulated phase change material with thermoplastic polyurethane
b. Sub-area:	Experimental Solar Thermal Engineering
c. Area Description:	The shape stabilization of microencapsulated phase change materials with enhanced thermal properties in thermoplastic polyurethane for a composite solar thermal energy storage material with minimal leakage.
d. Aims & Objective (in Bullets):	Aim: The development of a novel composite for thermal energy storage by incorporating the microencapsulated phase change material with thermoplastic polyurethane Framed Objectives are:

- To develop paraffin/melamine-formaldehyde(MF) microencapsulated phase change materials with different types of thermal conductive co-fillers such as magnetized Ni-GO; expanded graphite (EG), and graphene oxide (GO) by in-situ polymerization technique.
- To develop a shape stabilized composite PCM (ss-cPCM) based on TPU and microencapsulated PCM with co-filler.
- To design and fabricate the thermal storage experimental setup for the developed shape stabilized composite PCM.

e. Duration (MM/YYYY):

02/ 2022 TO 08/ 2022 (6 months)

f. Project Summary (1000 words):

Thermoplastic polyurethanes (TPU) are recyclable materials with fewer changes in their thermal characteristics. They have unique tailorable properties due to the block copolymer chain's hard and soft segment interactions. Even though their potential as a supporting matrix for shape stabilizing the phase change materials is not studied yet. The microcapsules' solar thermal energy storage efficiency has been improved by its shape stabilization with the TPU matrix that offers both support and unique tailorable property. The final material is called shape stabilized composite phase change material (ss-cPCM). Therefore, this research aims to develop an environment-friendly energy harvesting material based on thermoplastic waste and solar energy. The novel ss-cPCM is developed from thermoplastic polyurethane incorporated with microencapsulated phase change material/co-fillers by the chemical processes followed by the solution casting method. The different types of thermal conductive co-fillers enhanced the in-situ polymerization technique prepares paraffin/melamine-formaldehyde microcapsules.

In this research, highly thermal conductive nanoparticles based on graphene such as magnetized graphene oxide; graphene oxide, and expanded graphite are selected as the co-fillers added at different concentrations from 0.5,1.0, and 1.5mg/ml. The uniform aqueous dispersion of the different co-fillers at varying concentrations is added to the in-situ polymerization mixture. The

core phase change material is paraffin and the shell polymer is melamine -formaldehyde in this research. The clear and transparent melamine-formaldehyde prepolymer solution for in-situ polymerization should be prepared separately only by optimizing a reaction. The controlled and optimized conditions of in-situ polymerization give out a microencapsulated phase change material solution; in turn, vacuum drying gives smooth and uniform microcapsules.

The morphological and thermal characterization of microcapsules will be done by scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), Fourier transformation infrared spectroscopy (FTIR), X-ray diffractometry (XRD), thermogravimetric analysis (TGA), differential thermal analysis (DTA), differential scanning calorimetry and the PCM leakage test. These characterization results will be helpful to the selection of the best co-filler/MPCM combination and therefore it can be subjected for shape stabilization with the selected matrix, the thermoplastic polyurethane. The performance analysis of the microcapsules as thermal energy storage material will be done in the designed heat sink experimental setup, which should be given for fabrication later.

The microencapsulation technique effectively prevents the phase change material leakage during phase transitions but still exists the same at the end level usage. So, there is a need for shape stabilization of the microcapsules with a supporting matrix. As thermoplastic polyurethane has a lot of unique physical and chemical properties, it is not explored as a supporting matrix for microcapsules. Even though the supporting materials don't have any role in the heat storage capacity of the material, they can enhance the shape stabilization of the material, thereby increasing the heat transfer rate through the material. The solution casting method also successfully performed preliminary studies to develop the ss-composite film incorporated TPU with different concentrations of microcapsules. But the process optimization based on the characterization results only can give an idea about better consistent film composite. So, there is a need for detailed experimental study for the development of an ss-cPCM for a novel thermal energy storage material with minimal leakage and outstanding performance.

The preliminary experimental setup required for the performance analysis of the shape stabilized microcapsule composite film is made ready to use in the laboratory itself. A more detailed and sophisticated setup should be designed and fabricated based on these experimental results. This research since focused on solar renewable energy storage with the thermoplastic polyurethane material, which is not yet explored, surely lightens the path of future researchers towards an eco-friendly novel composite with competitive results and opportunities.

g. Technical Details

a. TRL

This research satisfies the technology readiness scale criteria from T1, T2, T3, T4, T5, and T6.

b. Methodology

1. Preparation of conductive co-fillers

The different graphene-based thermal conductive co-fillers are magnetized graphene oxide, expanded graphite, etc. Chemical reduction processes prepare the magnetized graphene oxide; the graphene oxide is synthesized by modified hummer method and the intercalation processes prepare the expanded graphite. All the processes are done under controlled temperature conditions. Fig1 gives the flowsheet for the corresponding method.,fig2. and fig 3. respectively.

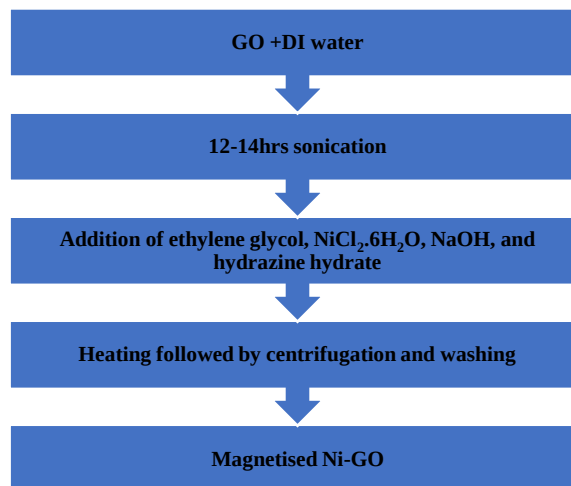


Fig.1. Preparation of magnetized graphene oxide by chemical reduction processes



Fig.2. Preparation of graphene oxide by modified hummer method

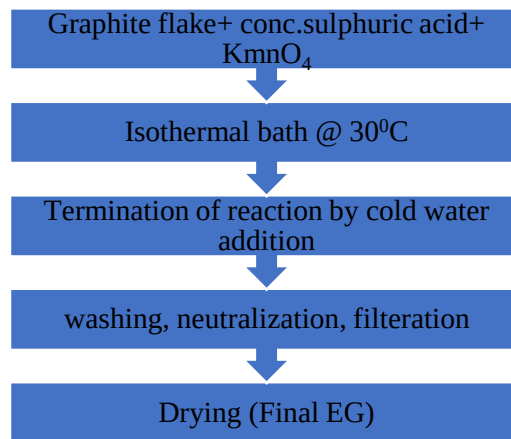


Fig.3. Preparation of expanded graphite by intercalation of expandable graphite

2. In-situ polymerization

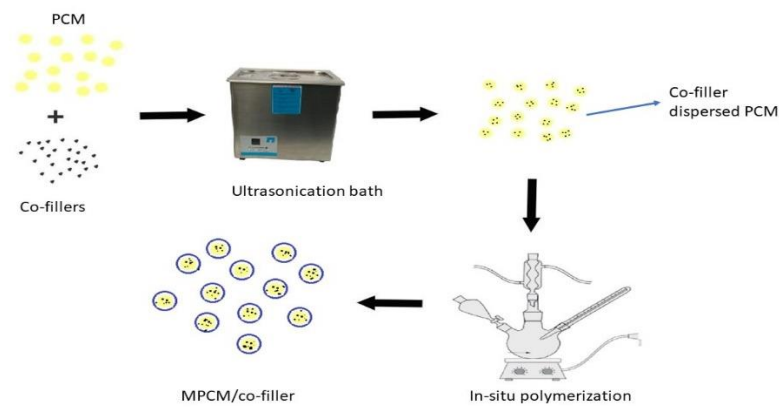


Fig.4. Preparation of paraffin-cofiller/melamine-formaldehyde microcapsules by in-situ polymerization technique

3. Development of shape stabilized composite PCM with TPU by solution casting method

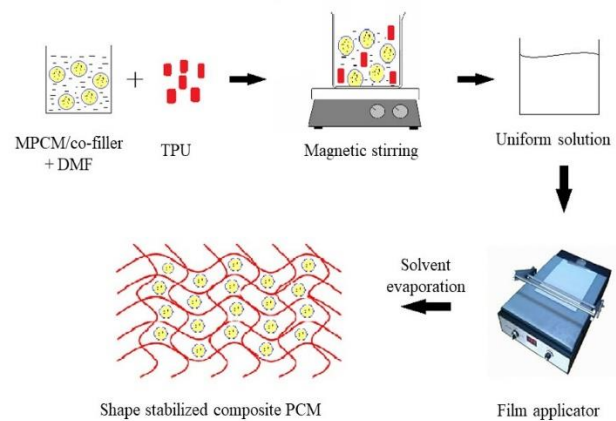


Fig.5. Shape stabilization of MPCM/co-filler into TPU

c. Design & development

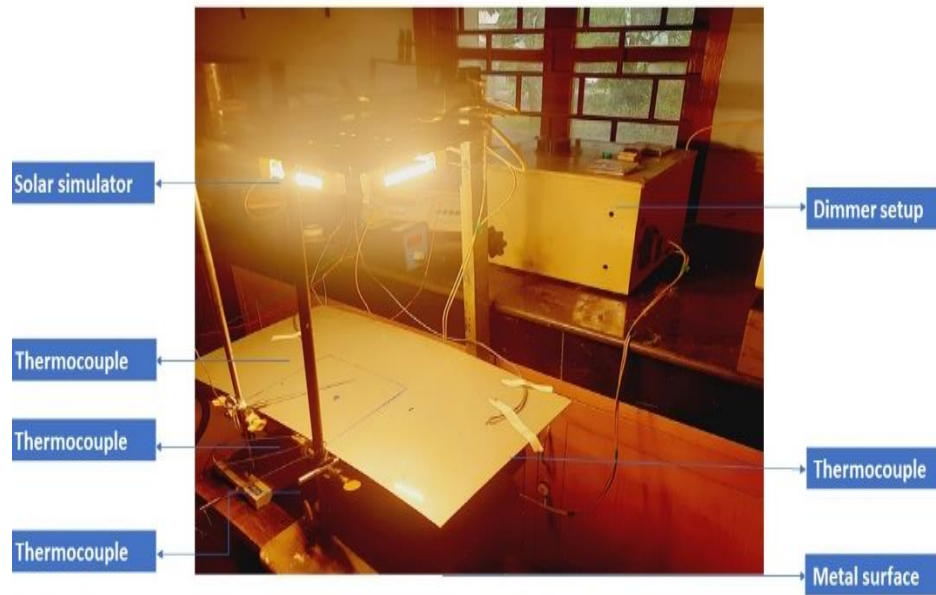


Fig.6. Experimental setup for thermal energy performance of ss-PCM

The flowsheet for the overall methodology of the research is given below:

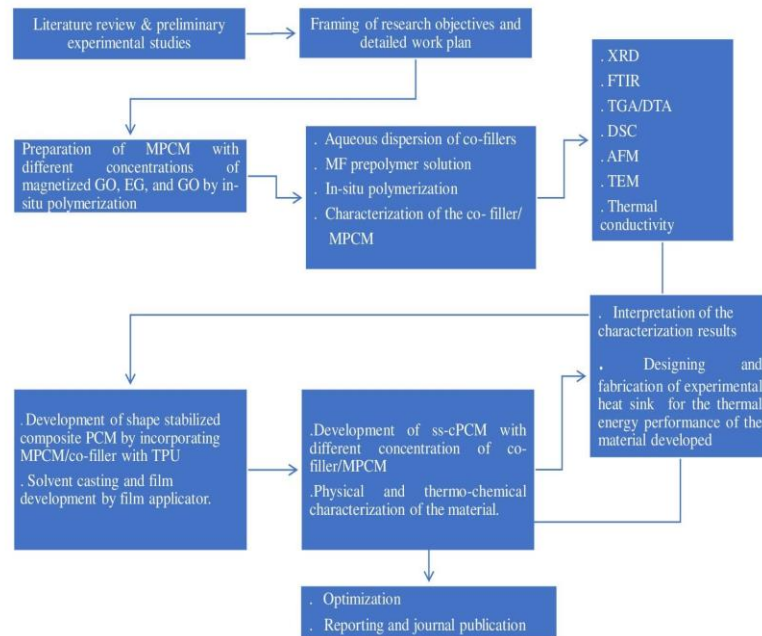


Fig.7. The overall methodology of the work

d. Facilities and Instrumentation required

1. Ultra sonicator
2. Magnetic stirrer
3. Overhead stirrer
4. Thermostat water bath
5. Ph meter
6. Vacuum filter
7. Hot air oven
8. Vacuum Drier
9. Film Applicator
10. Solar simulator
11. Thermocouples
12. DAQ
13. Computer

e. Consumables (Scientific materials)

1. Paraffin wax (water content less than 3%)
2. n-octadecane
3. Sodium hydroxide (pellet form, NaOH, molar mass = 40.00 g/mol,)
4. Urea (crystalline form, $\text{CH}_4\text{N}_2\text{O}_5$ molar mass = 60.03 g/mol,)
5. Ammonium chloride (NH_4Cl , molar mass = 53.49 g/mol, crystalline state)
6. Deionized water
7. Resorcinol (crystalline form, $\text{C}_6\text{H}_4(\text{OH})_2$, molar mass = 110.11 g/mol)
8. Melamine (extra pure)
9. Formaldehyde solution (molar mass 30.03 g/mol)
10. Acetic acid glacial
11. Sulphuric acid
12. Phosphoric acid
13. Hydrochloric acid
14. Potassium permanganate
15. Ethylenediamine
16. N-N-dimethyl formamide
17. Sodium lauryl sulfate
18. pH strip
19. Whatman filter paper
20. Petroleum ether solvent
21. Photo-initiator ferric chloride
22. Nickel chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)
23. Polyethylene (MFI: 25 g/10 min)
24. PEO solvent (MV: 400,000 g/mole)
25. Thermoplastic polyurethane
26. Graphene oxide
27. Expanded graphite
28. Synthetic graphite

h. Expected output and outcome

- Solar thermal energy storage in coatings.
- Improving thermal stability and reliability of MPCMs by incorporating them into a supporting matrix.
- Scope of recycling thermoplastic materials towards a value-added product.
- Eco-friendly end level uses of the composite for thermal comfort and smart thermal products.
- Applications in thermal conductive films.
- Heat transfer fluid in solar thermal energy storage units.

i. Industry/Institute Collaboration- NIL

j. Funding required from I-STEM (with justification:

Details of Requirements & Mode of the Engagement: (Attach the quotation for the consumables)

i. Short term Engagement: **6 months**

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

i. Testing Charges- Rs. 116850 /-

Sl.No.	Characterization Mode	Institute	Charge per sample	Total cost
1.	FESEM	Amrutha Institute of medical science, Kochi, Kerala	1000/-	15000/- (15 samples)
2.	FTIR	NITC, Kerala	50/-	1000/- (20 samples)
3.	DSC	NITC, Kerala	350/-	7000/- (20 samples)
4.	XRD	NITC, Kerala	200/-	4000/- (20 samples)
5.	AFM	IISER, TVM, KERALA	2000/-	20000/- (10 samples)
6.	HRTEM with SAED	STIC, CUSAT, Kerala	6785/-	67850/- (10 samples)
7.	TGA/DTA	NITC, Kerala	NIL	NIL
8.	Contact angle	NITC, Kerala	NIL	NIL
9.	Raman Spectroscopy	NITC, Kerala	200/-	2000 (10 samples)

ii. Consumable charges -**NIL**

iii. Travel Cost (Boarding and Lodging, if any)- **Rs.3000/-**

b. Usage of fabrication and testing facilities across the country (Fabrication Mode)-**Not required**

i. Fabrication and testing Charges

ii. Consumable charges (if any)

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

(i) Medium-term Engagement: NA

a. Execution of project in any one of the Host Institution (Project Mode)

a. Fabrication and or testing Charges

b. Consumable charges (if any)

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

b. Execution of project in Multi-Host Institutions (Project Mode)

a. Fabrication and or testing Charges

b. Consumable charges (if any)

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

(Round-trip to the Host Institution by 2nd class (Non-AC) railway via the nearest route)

Details of User's Bank Account:

(Please send a copy of the canceled cheque/copy of the passbook carrying all the information needed)

Beneficiary Name (User's name): **Aiswarya V** PAN: AVQPV4703L

Bank Account No. **67282745400**

Name of the Branch: **SBI, Ambalathara, Trivandrum, Kerala.**

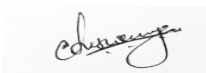
IFSC Code: **SBIN0071191**

Name (Research Scholar): Aiswarya V

Name (Guide): Dr. Sudev Das

Assistant Professor, CHED, NIT Calicut

Signature:



Signature:



22.02.22

Note: Proposed proposal must be executed within stipulated duration (max. 6 months) to avoid fund availability issues. The 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 the I-STEM portal only along with a copy of the travel forms and original tickets through the post to the Nodal Officer, I-STEM, Indian Institute of Science, Bangalore. A scanned copy of all the documents may be sent in advance.