SOCIETY OF PHYSICS STUDENTS
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SPS Chapter Research Award Proposal

Project Proposal Title	Microencapsulated Thermochromic Materials for Energy Saving Applications
Name of School	Florida Polytechnic University
SPS Chapter Number	2054
Total Amount Requested	$2000.00

Abstract

This proposal's aim is to develop and demonstrate microencapsulated thermochromic materials for energy saving applications. These microencapsulated thermochromic materials have wide applications in energy storage, buildings, textiles, antiques, paints, and pigments. The project will focus on the synthesis of novel, encapsulated thermochromic materials. In addition, the physico-chemical characteristics of the materials will be investigated with state-of-the-art tools to understand the surface morphology, phase transition behavior, and effectiveness of the encapsulant on the stability of the incorporated thermochromic dye.

Proposal Statement

Overview of Proposed Project

- **Research question** – What question does the research aim to address?
  Thermochromic materials (TCMs) possess color change properties with respect to increase of temperature. TCMs often degrade under the exposure to sunlight with a wide range of wavelengths from UV to near IR. Due to this degradation, TCMs usage for potential applications such as coating exteriors of the buildings and energy storage are limited. The research question currently under investigation can be stated as follows. Are there any potential methods to protect these organic TCMs from photo and/or thermal degradation while maintaining their thermochromism and maximizing their utilization in these varied applications.

- **Motivation** – Why is this research project important? What are some potential impacts of the research?
  This research project is very important to understand and develop the microencapsulation process that enables one to prevent the degradation of core TCM particles which are often exposed to the sun's radiation. Some other potential impacts of this research are to create novel materials for thermal storge and energy savings in buildings.

- **Brief description –** What will the research project entail?
  The research project entails the synthesis and development of microencapsulated TCMs via the core-shell nanocomposite materials. The core is the TCM while the shell materials are based on the inorganic metal oxides. Analytical characterizations such as x-ray diffraction, scanning electron microscopy, and energy dispersive spectroscopy will be carried out to study the structural, microstructural, and compositional features of the microencapsulated TCMs.

- **Research goals of the project –** What will the research project accomplish?
  This research project will develop novel microencapsulated TCMs that can be potentially used for energy storage and energy saving applications. The overarching goals of the project is to learn how to synthesize and characterize the novel microencapsulated TCMs using the state-of-the-art tools.

- **SPS connection –** How will this activity strengthen the objectives of the SPS program, both at the proposing school and nationally?
  This chapter's research activity strengthens my undergraduate research experience and helps the SPS Florida Poly chapter grow in terms of publication output (SPS Observer, Journal of Undergraduate Research), STEM outreach, and related endeavors. This chapter research award and experience will enable me to have research experience that will help me to qualify for summer internships at the SPS sponsored National Laboratories or Universities.

Background for Proposed Project

The technology of chromaticity in devices can be used to vary the throughput of visible light and solar energy for windows and buildings surface application. The technologies can make use of a range of *chromic* materials either by themselves or in combination with other encapsulants [1]. The rapid development of smart materials has encouraged researchers to explore new possibilities. The market for chromic materials is growing rapidly because of their optical, storage and color changing properties under different stimuli. Examples of such materials are thermochromic inks [2], electrochromic windows [3], and photochromic fibers [4]. Although different chromic materials change color by different stimuli, the mechanism for such changes is similar, that is, a reversible electron or ion transfer. Generally, electrons need energy to overcome a potential barrier (ΔE) to complete the transfer and this can be provided by photons, heat, or electric potential. The TC chemicals also need to be metastable for the process to be reversible [5]. Leuco dye systems contain three components: dye, developer, and long-chain solvent. Briefly, the dye interacts with the developer to form one color when the solvent is in the solid form, while upon heating and melting of the solvent, this interaction is lost, forming an alternate color. Thus, the color change of the leuco dye system is controlled by the melting or crystallization of the long-chain solvent [6].

According to Nassau, a dye can undergo photodegradation under sunlight when ultraviolet light interacts with the chemical bonds and destroys the chromophores in the dye [7]. Zhang et al. reported that the encapsulation of a thermochromic material (TCM) by amorphous silica could protect it from UVB light (290-320 nm) [8]. Therefore, it is important to encapsulate the TCM with an inorganic metal oxide. A variety of metal oxide nanoparticles (NP's) such as SiO_2, Al_2O_3, Fe_3O_4, ZrO_2 and TiO_2 [9-13] have been used for surface modifications and coating. Among them, the addition of nanometer sized SiO_2 has received the greatest attention because of its physical and chemical stability, and wide availability. However, addition of nanosized silica with the nanosized titania gives rise to the additional effect of photocatalytic properties due to the presence of titania [14]. For this the Korean workers have developed nano hollow-spheres of the combined silica-titania systems which may be a potential approach for encapsulation of TCM. However, in our present work, we are mainly focusing on the encapsulation of TCM by the metal oxides, for example, SiO_2, ZnO etc. Extensive characterization and color changing behavior will be systematically carried out for the microencapsulated TCMs.

Expected Results

Themochromic color change characteristics of the microencapsulated TCMs are expected to show the transition of color from blue or black to white at around 35 ^{o}C. This phase transition of the TCM dye will be recorded by Differential Scanning Calorimetry (DSC) to estimate the enthalpy of reaction during endothermic and exothermic processes. The x-ray diffraction studies will be carried out and it is expected that the crystallinity of the shell material such as an inorganic metal oxide will be determined. The scanning electron microscopy and energy dispersive spectroscopy studies are expected to reveal the core-shell microstructures of the microencapsulated TCMs with compositional weight percentages of the constituent elements. Preliminary results of the TCMs micrographs show the spherical particles as given in Figure 1.

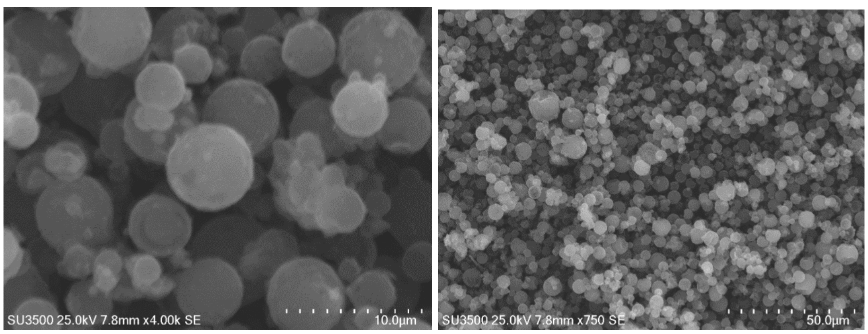

Figure 1: SEM micrographs of TCMs at different Magnifications

Description of Proposed Research - Methods, Design, and Procedures

Methods, Design, and Procedures

Materials

The commercial Leuco dye that changes color at about 30^{o} C will be obtained from Amazon Company. The blue dye changes color at about 35^{o} C and contains 1-tetradecanol, crystal violet lactone (CVL) and bisphenol A (BPA), the surfactant sodium dodecyl benzene sulfonate (SDBS), the precursor tetraethyl orthosilicate (TEOS) and encapsulation precursor catalyst, ammonia, which will be procured from Sigma-Aldrich and used without further purification.

Microencapsulation of commercial dye by SiO_2 nanoparticles

For the preparation of commercial dye encapsulated by SiO_2 nanoparticles, 80ml of DI water is measured and taken in a beaker. It is kept for stirring on hot plate at 400 rpm and at 70 ^{o}C. Then 0.5 g of commercial Leuco dye is added to the beaker. After 5 minutes, sodium dodecyl benzenesulfonate (SDBS) and tetraethyl orthosilicate (TEOS) are added to the solution and stirred until an emulsion is formed. Then ammonia is added as a catalyst and kept for stirring for aan additional 2 hours. Finally, the sample is washed thoroughly with ethanol and kept for drying in an incubator at 150 ^{o}C for 24 hours. The microencapsulated TCM powder sample is stored in a glass tube for further characterization and property measurements.

Analytical Characterizations

The Hitachi SU3500 is used to obtain a high-resolution scanning electron microscopy (SEM) image. The elemental analyses of both commercial dye based, and blue dye samples are characterized by energy dispersive x-ray spectroscopy using Oxford Instrument's EDAX detector attached to SU3500 SEM, with a working distance of 10 mm, a takeoff angle of 35^o and an accelerating voltage of 15 kV. The Hitachi's HT7800 series is a HRTEM instrument used to determine, if the encapsulation is successful because it can offer a higher resolution to allow the delineation of the sample's surface morphology. Dynamic Light Scattering (DLS) will be ran on samples to see the extent that the reaction schemes can break down large aggregates of the TCM samples using the Zetasizer Nano Series (ZS, Model: ZEN3600) DLS which is designed to measure particle size, molecular weight and zeta potential. Fourier transform infrared spectroscopy (Agilent 630 FTIR) will be utilized to determine the chemical and bonding environment. For the FTIR experiments, an ATR cell will be used for the plain dye and core@shell powder samples placed on the diamond window and applying pressure using a screw gauge. The transmission mode will be used to collect IR signals between 400 to 4000 cm^{-1} wavenumbers. The thermal properties of these SiO_2 and inorganic encapsulated TCMs will be analyzed by differential scanning calorimetry (TA Instruments, DSC 2500) and thermo-gravimetric analysis (Mettler Toledo, TGA/DSC 1 $STAR^e$ Systems). For the TGA studies, the TCM@SiO_2 sample size of <10 mg will be heated from room temperature to 600 ˚C with a ramping rate of 10 ˚C/min under nitrogen flow of 50 mL/min. To determine the visual color change, TCM@SiO_2 and other TCMs core shell systems will be placed on white filter paper on top of a hot plate. The hot plate's temperature will be adjusted in increments of 5 ˚C while the actual temperature of the samples will be determined by an infrared thermometer.

Plan for Carrying Out Proposed Project

This section should detail the plan for carrying out the project, in bullet or paragraph form. Include, at minimum:

- **Personnel**
  - Daniil Ivannikov – SPS Member, Full Time Undergraduate Research Student, Florida Poly
  - Dr. Sesha Srinivasan – SPS Member, Chair, Engineering Physics/Natural Science, FL Poly
  - Dr. Scott L, Wallen – Laboratory Research Staff, Natural Sciences Department, Florida Poly

- **Expertise** – Yes, Dr. Sesha Srinivasan and Dr. Scott Wallen are experts in materials' development and synthesis, analytical characterization studies and scientific publication preparation.

- **Research space** – We will conduct this proposed work in the wet chemical research laboratory of Florida Polytechnic University. We have a Characterization Lab where we will execute various characterization studies such as SEM, XRD, EDS, FTIR, UV-Vis, DLS, TGA/DSC and CIE Lab.

- **Contributions of faculty advisors or the department (equipment, space, etc.)**: Faculty Advisor Dr. Sesha Srinivasan and the Lab Research Staff Dr. Scott Wallen will provide their valuable time to design the project goals, and define the protocols for undergraduate research studies. Equipment such as synthetic reaction glassware and characterization equipment is housed at Florida Polytechnic University. We can also use our collaborator's research facility at the University of South Florida.

Project Timeline

Tasks	Start Date	End Date	Milestone/Activity
Task 1	1/1/2021	1/31/2021	Project Start, Literature survey, Purchase of chemicals
	2/1/2021	2/28/2021	Materials' synthesis and optimization (Wet chemical apprpoach)
Task 2	3/1/2021	3/31/2021	
	4/1/2021	4/30/2021	
Milestone 1	5/1/2021	5/31/2021	**Milestone 1: Interim report submission**
Task 3	6/1/2021	6/30/2021	Analytical characterization and property measurements
	7/1/2021	7/31/2021	
	8/1/2021	8/31/2021	
Task 4	9/1/2021	9/30/2021	Thermochromic materials testing and validation
	10/1/2021	10/31/2021	
	11/1/2021	11/30/2021	
Milestone 2	12/1/2021	12/31/2021	**Milestone 2: Final report submission**

Budget Justification

Equipment: The project scope is centralized to study the color change characteristics of the thermochromic materials. For this study, a CIE Lab Color Meter is essential and an integral part of the project's equipment acquisition. The color meter from PCE Instruments CSM 3 is the ideal measuring device for application in quality control and offers a high accuracy. This color meter provides the CIE Lab values which will enable us to calculate the color coordinates and quantify the color change properties of microencapsulated TCMs. The CSM 3 with the price tag of $1,261.00 (quote attached) is budgeted.

Chemicals/Solvents/Gases and Supplies: A number of chemicals including TCM commercial dye, metal oxides such as SiO_2, ZnO, and TiO_2, solvents, surfactants, acids, and bases will be procured for the development of microencapsulated TCMs. Gases such as nitrogen and compressed air will be procured for the TGA measurements and other synthesis purposes. Supplies such as TGA pans, and accessories, weighing boats, Whatman filter paper are budgeted (total $739.00) for the proposed research work.

Total budget of $2,000 is justified above for the successful completion of the proposed project.

In-Kind Support: Glassware, DI water, state-of-the art instruments, expert research personnel and their time (my mentor, laboratory staff and other senior researchers) will be extensively available to support this project. Our collaborator from a Research One Institution, the University of South Florida and an international collaboration with the University of New South Wales, Australia are available for technological discussions and knowledge transfer as well as use of their research facilities.

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