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Revolutionizing Engineering Education: Bridging Theory with Practice through Microfluidics and Material Characterization

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Revolutionizing Engineering Education: Bridging Theory with Practice through Microfluidics and Material Characterization

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Abstract

This article presents an innovative instructional approach to revolutionize engineering education by integrating microfluidic devices and material characterization tools. Focused on key engineering principles such as thermodynamics, heat transfer, and crystallization, this method offers students a dynamic, hands-on learning experience. It emphasizes the potential of microfluidic devices to manipulate small fluid volumes, highlighting their capacity to enhance heat and mass transfer, accelerate reaction kinetics, and reduce reagent consumption. With applications across disciplines like biology, chemistry, medicine, climate science, and engineering, microfluidic devices serve as a versatile platform for experiential learning, bridging theoretical knowledge with real-world applications.

Concurrently, material characterization tools measuring physical and chemical properties, including phase transitions and enthalpy, complement this instructional paradigm. The integration of microfluidics and material characterization enables students to grasp and apply fundamental engineering principles actively. Illustrated through a case study, students explore crystallization aspects in water enriched with nucleating agents using microfluidic devices. The device detects crystallization onset in water droplets, with freezing efficiency determined from images of the nucleation process against observed freezing temperatures. Comparative analysis with a differential scanning calorimeter reveals exothermic energy release during the water droplet's phase transition, providing a deeper understanding of thermal characteristics in crystallization.

This paper comprehensively outlines the approach's objectives, methodologies, and outcomes, positioning it as a transformative force in engineering education. By encouraging active exploration, critical thinking, and collaborative discourse, this innovative approach significantly contributes to the evolution of effective engineering education methodologies, preparing students for the challenges of a rapidly advancing technological landscape.

Introduction

In the rapidly changing landscape of technology, engineering holds a pivotal position, continuously adapting to meet the diverse demands of industries. Success in this field requires a strong theoretical foundation coupled with practical experience. While theoretical understanding forms the basis, engineering thrives on integrating practical skills with academic knowledge. Hands-on projects play a crucial role in enhancing critical thinking and problem-solving abilities, essential for tackling real-world challenges. Through these experiences, students develop the skills necessary for innovation and overcoming obstacles in their future careers. Recognizing the significant value of practical experience, it is essential to incorporate such opportunities into the engineering curriculum[1].

West Texas A&M University (WTAMU), located in the Texas Panhandle, distinguishes itself as an educational institution. As a Primarily Undergraduate Institution (PUI) and a Hispanic Serving Institution, WTAMU serves a diverse student population, including many first-generation college students. The College of Engineering at WTAMU offers a variety of disciplines and is supported by advanced research facilities like the Palo Duro Research Center. This research hub boasts cutting-edge equipment such as microfabrication and high-speed microscopic imaging tools, high-end material characterization tools such as scanning electron microscope (SEM), X-ray diffraction (XRD), Thermogravimetric analysis (TGA), differential scanning calorimeter (DSC), and a 3-flex surface analyzer enhancing students' research capabilities. Leveraging these resources, this article presents a case study illustrating the application of theoretical knowledge in practical projects, disseminated through seminars and conferences. Such initiatives enrich students' learning experiences, preparing them effectively for the multifaceted challenges in the engineering field.

The educational exploration delves into the core concepts of crystallization, the phase change of materials, and the energy dynamics inherent in any phase change. These foundational principles are integral to the curriculum across several engineering disciplines, including mechanical, civil, and environmental engineering, embedded within fluid mechanics, thermodynamics, and material science courses. To bridge the gap between theoretical knowledge and practical application, a project is designed to allow students to implement their theoretical understanding of material phase change using two industry-relevant tools: microfluidic devices and a differential scanning calorimeter (DSC).

Microfluidic devices, heralded as revolutionary in engineering education, grant students precise control over fluid dynamics at the microscale. These devices manipulate small fluid volumes, showcasing their potential to enhance heat and mass transfer, accelerate reaction kinetics, and reduce reagent consumption[2, 3]. With applications across disciplines such as biology, chemistry, medicine, climate science, and engineering, microfluidic devices emerge as a flexible platform for experiential learning, effectively connecting theoretical knowledge with real-world applications[4]. Similarly, material characterization tools like the DSC unveil exothermic energy release during the phase transition of a material. Determining the onset and endset of the freezing point from the phase transition curve, these instruments provide students with a hands-on platform to delve into and apply theoretical concepts[5].

Water is chosen as the phase change material for the project, given its ubiquitous presence and extensive use across industries. The exploration of ice nucleation in liquid water holds significance across diverse scientific and technological fields, including determining the fundamental physicochemical properties of water, atmospheric phenomena, and applications in food[6, 7], pharmaceuticals, and cryobiology[7]. The study delves into the two main pathways of water droplet freezing: homogeneously[8] and through heterogeneous nucleation catalyzed by ice nucleating particles (INPs) or other surface interactions[9-11]. A pivotal aspect of this exploration involves distinguishing between homogeneous and heterogeneous crystallization processes, forming the basis for understanding material behavior.

Illustrated through a case study, students employ microfluidic devices to explore homogeneous and heterogeneous crystallization aspects in pure water enriched by nucleating agents. The microfluidic device detects the onset of crystallization in water droplets, and freezing efficiency is determined from images capturing the nucleation process as a function of the observed freezing temperature. A complementary analysis facilitated by the DSC reveals exothermic energy release during the phase transition of the water droplet, offering insights into thermal characteristics inherent in the crystallization process.

This study delves into the methodologies, outcomes of experiments, and student learning outcomes utilizing Microfluidic Devices and DSC to discern between homogeneous and heterogeneous crystallization processes. Case studies and experiments are presented, emphasizing the practical implications of understanding and manipulating these crystallization phenomena. The aim is to showcase the transformative impact on students' comprehension of material characteristics and behavior, highlighting the effectiveness of engaging students in microfluidics and material characterization research within the engineering curriculum. The proposed strategy is positioned as a transformative force in engineering education, contributing significantly to the ongoing evolution of effective methodologies. By fostering an environment that encourages active exploration, critical thinking, and collaborative discourse, this innovative approach sets a new standard for immersive and engaging engineering education, preparing students for the challenges of a rapidly advancing technological landscape.

Methodologies

The investigation project focused on water's homogeneous and heterogeneous crystallization and phase change processes, structured into i) a Microfluidic Module and ii) a Differential Scanning Calorimeter Module. Both modules encompassed sample preparation, experimentation, and comprehensive data acquisition and analysis. The execution of both modules was conducted by two graduate students and one undergraduate student. Below is a summary of the activities conducted under each module.

i) Microfluidic Module

This module was designed to explore the onset temperature and freezing efficiency of both homogeneous and heterogeneous nucleation of water. The following student learning activities were undertaken:

Design and Fabrication of Microfluidic Devices: The microfluidic device was meticulously designed using AutoCAD, drawing inspiration from previous research by Bithi et al.[12, 13]The design employed the principle of flow resistance to establish a static droplet array (Figure 1). The device featured interconnected droplet parking traps with a nanoliter volume. The fabrication process followed standard soft lithography[14], where a master mold was created on a silicon wafer through photolithography. Subsequently, a Polydimethylsiloxane (PDMS) replica of the device was generated using the master mold. The resulting PDMS replica underwent cutting, and inlet and outlet reservoirs were defined by punching holes. This replica was then placed on a PDMS-coated glass slide, partially cured, and further cured to establish an irreversible seal. This detailed process enabled the production of multiple devices for experimental purposes.

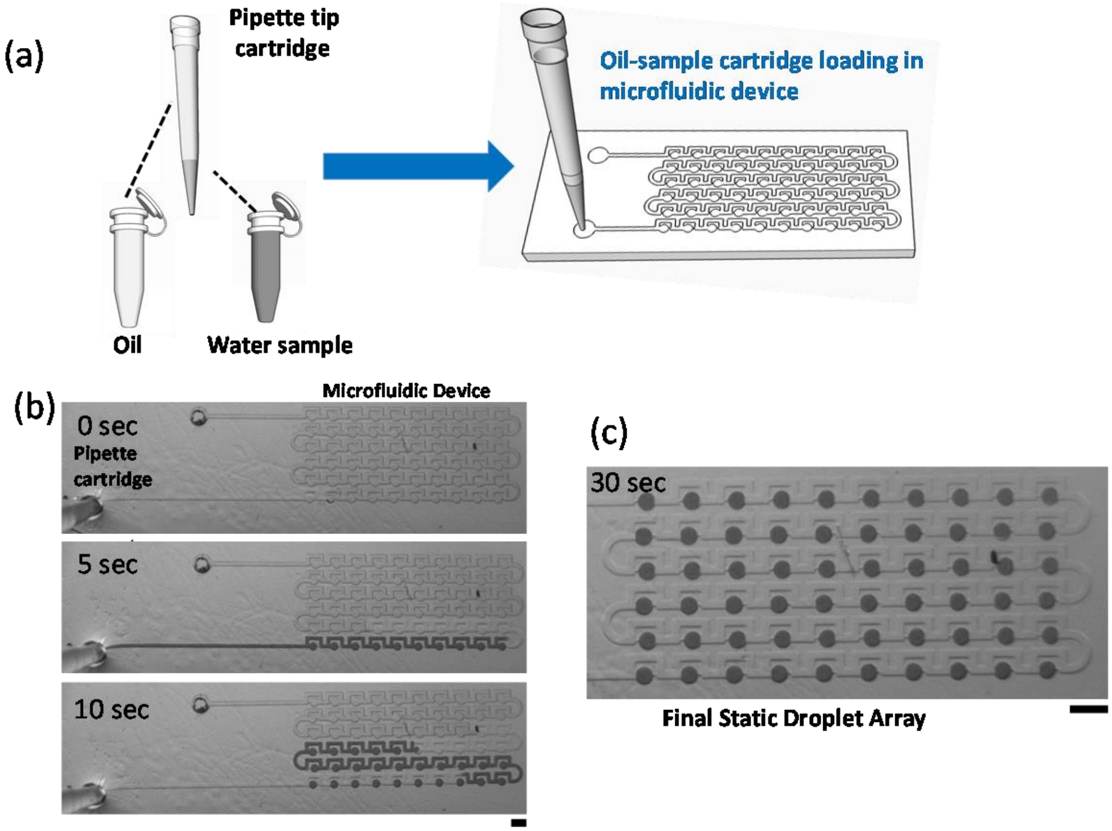


Figure 1. Sample loading and trapping in a microfluidic device using oil and water samples. a) A cartridge of water sample and mineral oil is created in the pipette tip and loaded to the inlet of the device. b) Time-stamped images of plug motion through the device. The sample plug (dye solution) fills the channels and traps and is sequentially digitized by the oil plug producing an array of static droplets of uniform size. c) Final static droplet array. Scale bars are equivalent to 1mm.

Sample Preparation and Loading Process in Microfluidic Device: In the microfluidic module, the sample preparation and loading process aimed at investigating homogeneous and heterogeneous ice nucleation processes employed distinct techniques. High-performance liquid chromatography (HPLC) water served as the medium for homogeneous ice nucleation, while heterogeneous ice nucleation was assessed using 0.1wt% solutions formed by blending particles into HPLC water. Two commercially available particles, Snomax®[15] and illite NX[16], were utilized as ice nucleating agents.

A unique method of hand pipetting[17] was employed to create an array of ice-nucleating particle (INP)-laden nanoliter-sized droplets in the microfluidic device. Figure 1 illustrates the preparation process: a cartridge was assembled by sequentially aspirating 2μL of a water sample and 5μL of mineral oil. This cartridge was then loaded into the inlet of the microfluidic device. Time-stamped images tracked the motion of the plug through the device, resulting in an array of static droplets of uniform size. The microfluidic device's geometry dictated the mechanism of sample digitization, with the plug containing a dye solution and filling the channels and traps. This sequential digitization by the oil plug generated a consistent array of static droplets in under 30 seconds.

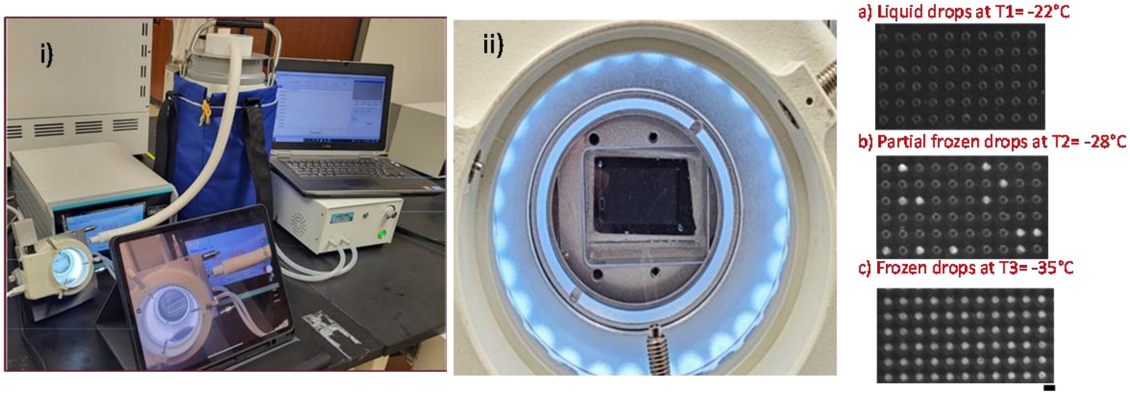


Figure 2. Ice nucleation experiment. i) Experimental setup for ice nucleation in microfluidic module. ii) Ice nucleation experiment in a microfluidic device. Snapshots (a-c) showing different stages of ice nucleation process with HPLC water.The scale bar represents 1mm.

The microfluidic device was positioned on a commercialized cooling unit (Instec, Inc., HCS321Gi) for visualizing and characterizing freezing events of individual droplets (Figure 2 i). This cooling unit facilitated the simulation and investigation of the immersion freezing of water and/or any INP-involved suspension, down to the homogeneous freezing temperature (below -35 °C), across a wide range of cooling rates from 0.01 to 30 °C per minute.

Data Collection and Analysis: In each experimental trial of the nucleation process, a temperature-controlled stage maintained a constant cooling rate of 1.0 °C min-1 until reaching a temperature of -50 °C. The freezing process was recorded at a rate of 30 frames per second (fps) using an iPad Pro over 45 minutes (from 5 °C to -42 °C). Images acquired during the nucleation process were analyzed to determine freezing efficiency as a function of the observed freezing temperature of the droplets. Initial manual detection of frozen droplets based on their contrast in grey values, as shown in Figure 2 (a-c) to temperature, was followed by a comprehensive analysis at 30-second intervals.

The frozen fraction (F_frozen) was calculated using the formula F_frozen = number of frozen droplets/total number of droplets at each temperature interval. Experiments were systematically replicated to ensure result consistency, eliminate potential errors, and enhance the findings' robustness. Multiple iterations of water samples were examined in each experimental run, revealing significant distinctions between tap water and HPLC water samples, including variations in freezing onset temperatures.

ii) Differential scanning calorimeter (DSC) module

The ice nucleation process is conducted using Differential Scanning Calorimetry (DSC) for an identical set of samples. Figure 3 is showing a skematic of DSC experimental setup. Two platinum crucibles were cleaned with ethyl alcohol prior to experimentation. A balance is zeroed out using the crucible and cap. A thin coat of pure petroleum jelly is applied to the inner base for the sample crucible. A pipette is used to dispense a single 3 μL droplet of solution in the center of the crucible. The platinum cap is placed on top of the crucible. The mass of the sample is measured and should be approximately 3 mg with an error of ±0.1 mg. The crucibles are placed within the DSC chamber. The DSC is pre-cooled to 5°C and is set to cool at 1°C/min to -45°C. The data is recorded and saved through the computer that controls the DSC software. Throughout this process, the DSC records the exothermic energy release during the phase transition of the water droplet. The onset and endset of the freezing point are determined from the resulting phase transition curve.

DSC serves as a valuable tool for measuring the heat flow associated with physical changes in materials. The DSC experiments provide comprehensive information about melting,

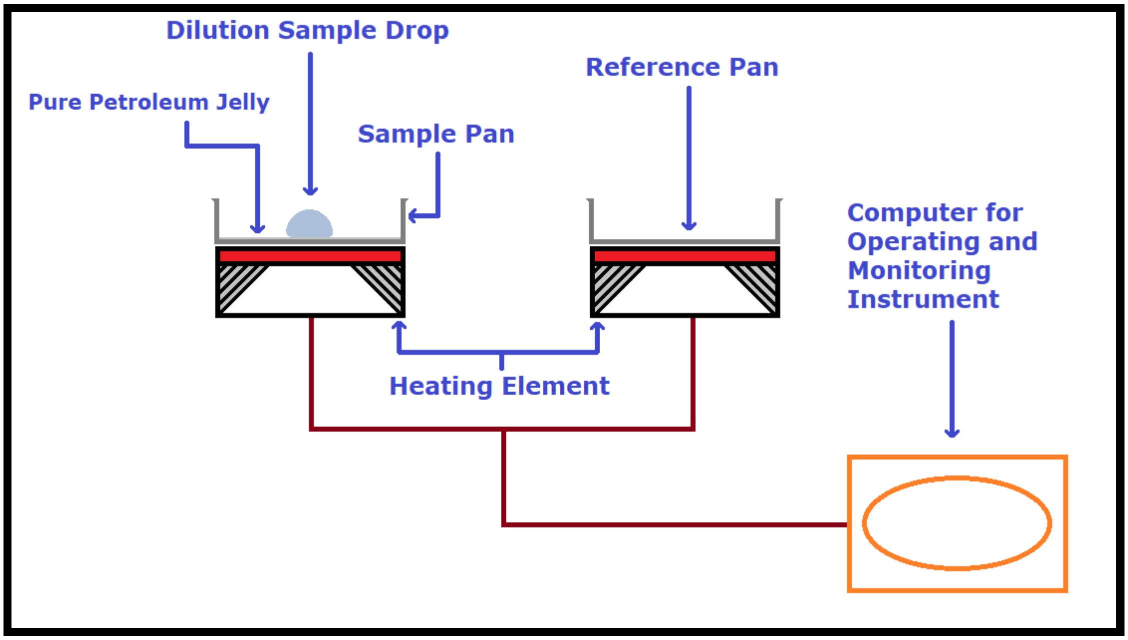


Figure 3. Experimental setup for ice nucleation in DSC module.

crystallization, and heat of fusion. The DSC instrument comprises sample and reference crucibles, both subjected to the same heating or cooling rate. The heat flow difference between the sample and reference is meticulously recorded, offering insights into the heat absorbed or released during various material changes. By employing DSC, students gain the ability to observe, analyze, and quantitatively assess thermal alterations in materials.

Experimental Outcomes and Discussion

Figure 4 encapsulates the freezing efficiency data derived from the microfluidic module. The frozen fraction of water droplets, including HPLC water and water samples with two distinct particles (illite N.X., and Snomax®is graphed as a function of temperature.

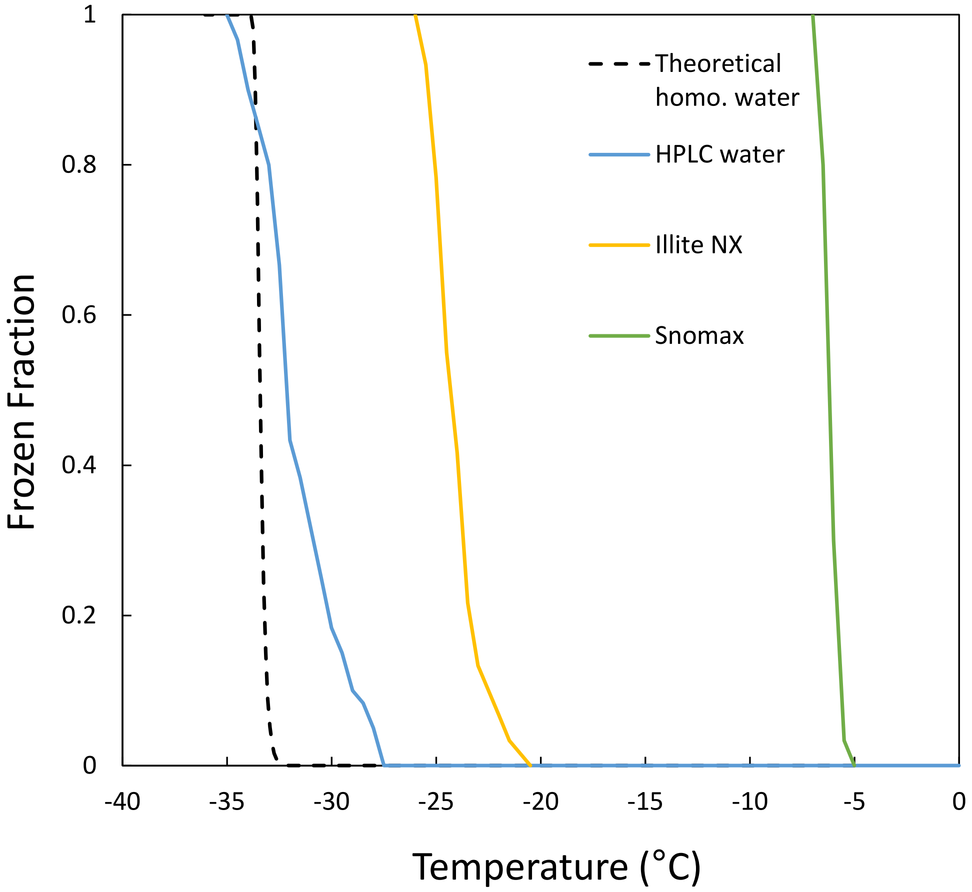


Figure 4. Results of Frozen Fraction (FF) spectra for homogeneous nucleation for high-performance liquid chromatography (HPLC) water sample, and heterogeneous nucleation as a function of temperature for water samples with Sanimax®, illite NX in Microfluidic module. The conceptual spectrum of a 15 nL of pure water droplet based on the classical nucleation theory superposed on the measured spectra. [8]

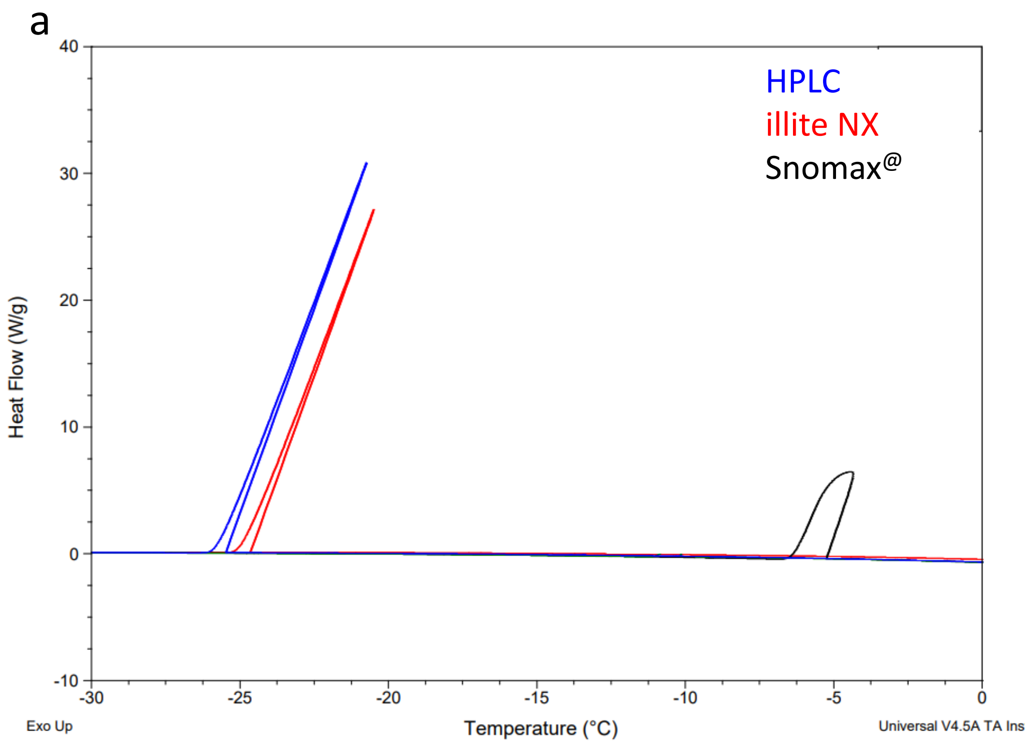
Overlaying the measured spectra with the theoretical spectrum of a 15 nL pure water droplet, based on classical nucleation theory[8], reveals characteristic temperatures corresponding to 50% frozen fraction (TFF50) values. The theoretical TFF50 values for pure water and HPLC water are approximately -33.4 °C and -32 °C, respectively. A comparison of HPLC data with the theoretical spectrum validates the microfluidic device's capability to reproduce quasi-homogeneous freezing. Discrepancies observed may stem from variations in stock tap water samples used for filtration or other factors influencing the sample preparation process.

Heterogeneous water nucleation is evident in water samples containing three ice nucleating particle (INP) proxies. Illite N.X., a mineral dust, initiates ice nucleation at a significantly higher temperature (-21 °C) compared to HPLC water. The TFF50 values for illite NX-water droplets are -24 °C, indicating heterogeneous nucleation due to the presence of mineral dust particles.

The second sample for heterogeneous freezing assessment is Snomax®, produced from Pseudomonas syringae bacteria with potent ice-nucleating proteins. These proteins aggregate into three types (Type I, Type II, and Type III) with varying freezing temperatures. Observations align with previous literature[15, 18], indicating ice nucleation of Snomax® occurs between -5.5 °C to -7 °C. This module successfully showcased the homogeneous and heterogeneous freezing behavior of HPLC pure water and different INPs, respectively.

Figure 5 provides insights into thermal properties from the DSC module, enabling students to observe and compare the thermal behavior of pure water and water with INP particles. Similar patterns of INP potential were observed in both modules, with Snomax® demonstrating the highest ice nucleating potential and illite N.X. exhibiting the lowest. However, characteristic temperatures corresponding to 50% frozen fraction (T_FF50) values in the DSC module were systematically higher than in the microfluidic module (Figure 5b). This disparity is attributed to the significantly larger drop volumes in DSC, potentially introducing more contamination in homogeneous freezing and providing more sites for heterogeneous ice nucleation. Students also calculated the heat of fusion from the area under the curves of DSC spectra for both homogeneous and heterogeneous ice nucleation processes (Figure 5b).

In conclusion, integrating microfluidic devices and DSC in this study showcased their potential in advancing experimental methodologies for studying ice nucleation and material characterization. These findings deepen our understanding of the phenomena under investigation and present promising avenues for further research.



b

Sample	Heat of Fusion	On-set of Freezing	End-set of Freezing	T_FF50
	J/g	˚C	˚C	˚C
HPLC	145.3	-27.13	-27.75	-27.26
Illite NX	245.5	-18.01	-19.03	-18.35
Snomax	295.1	-4.78	-6.87	-5.43

Figure 5. a) Example of calorimetric phase transition graph generated during the ice nucleation process in DSC module. b) Table contains the calculated values of heat of fusion, on-set and end-set teperatures of icecrystallization, and temperatures corresponding to 50% frozen fraction (T_FF50).

Student Learning Outcomes

Engaging in this project, which involved two graduate students and one undergraduate student majoring in Mechanical Engineering, provided valuable hands-on experience in soft lithography, microfluidic ice nucleation experimentation, DSC experimentation, and data acquisition and analysis. The exposure to practical applications heightened their interest in scientific endeavors and innovations and complemented their comprehension of theories taught in their courses. They showcased their project outcomes at conferences, including the 13th ICCPA International Conference on Carbonaceous Particles in the Atmosphere, the 18th Annual W.T. Faculty and Student Poster Session and Research Fair, and the American Geophysical Union (AGU) Fall Meeting. The project's impact has extended into ongoing academic pursuits, with one graduate student completing a master's thesis and initiating work as a research engineer at Colorado State University. Another graduate student is currently pursuing a master's thesis, drawing on the training acquired from this project. The undergraduate student has successfully earned a B.S. degree and is actively engaged in developing two research articles based on this project. A survey is also conducted among the participating students regarding their pre and post project learning in different topic areas as a result of engaging in research. Table 1 presents the results as percentages.

Table 1: Assessments of learning attained via engagement in research

Rating of Learning	Pre	Post
Skills in organizing and strategizing	66.66 %	100%
Supervising a group	33.33%	100%
Develop a hypothesis and test it	0%	66.66%
Samples handling, data collection, analysis	66.66%	100%
Problem solving	66.66%	100%
Creating presentation materials	66.66%	100%
Crafting summaries of research for publication	33.33%	100%

Conclusions

This article proposes a revolutionary instructional approach for engineering education by integrating microfluidic devices and material characterization tools. Focused on fundamental engineering principles, the strategy offers students hands-on experiences in thermodynamics, heat transfer, and crystallization. Microfluidic devices, with applications spanning various disciplines, enhance experiential learning by manipulating small fluid volumes. Integrating material characterization tools, particularly the differential scanning calorimeter, complements this approach, validating findings and providing more profound insights. Emphasizing active exploration and critical thinking, this transformative pedagogy sets a new standard, preparing students for challenges in an ever-advancing technological landscape.

Acknowledgments

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