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A Teaching-Learning Framework for Materials Characterization

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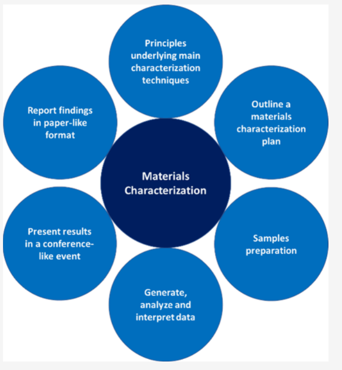
ABSTRACT: "Materials Characterization" is a broad discipline that plays a pivotal role in various scientific sectors and requires diversified training to provide learners with the necessary tools and skills for the investigation of materials' structure, microstructure, and properties. This study puts forward a comprehensive framework aimed at providing undergraduate STEM students with a wide range of competencies for material characterization. These include acquiring the essential theoretical knowledge of key characterization methods, the ability to construct experimental plans, the skills needed for sample preparation, and the aptitude to generate, analyze, and interpret data. By combining various strategies and pedagogical tools, the framework aims to facilitate self-directed and self-determined learning, allowing students to shape their educational journey and explore areas of personal interest within the discipline. Furthermore, the framework incorporates diversified approaches aimed at developing research proficiency and the ability to communicate research outcomes, both within conference contexts and through report formats resembling publications. The findings demonstrate the promising prospect that undergraduate students have the capacity to acquire the methodologies of scientists and to produce work of comparable quality. This study testifies the considerable potential that lies in engaging enthusiastic and capable students in scientific research and fostering the early development of future researchers.



KEYWORDS: *Materials Characterization, Teaching/Learning Framework, Active Learning, Research-Based Case Studies*

■ INTRODUCTION

The field of "Materials Science" has contributed to several advancements in science and technology, helping to transform lives throughout history.[1,2] "Materials Characterization" is widely recognized as a fundamental discipline that investigates the nature and properties of material systems,[3−6] based on continuously evolving techniques and advanced equipment available at universities and research centers worldwide.[5,6] Developments in the field have led to a demand for professionals with multidisciplinary competencies from various scientific backgrounds. The field expands beyond material science to encompass principles from solid-state chemistry and physics describing phenomena at different scales, from the macroscopic to quantum.[3,5] Accurate materials characterizations rely on the comprehensive understanding of the principles underlying the techniques, the ability to produce meaningful and reliable data, and the acumen in identifying artifacts for accurate interpretations.[7] Educators face significant challenges in creating professionals equipped with these competencies. The challenges may range from balancing theory and practice to resource constraints and complexity of the subject.[8] However, acquiring these skills remains crucial for driving advancements in scientific and technology sectors. Graduates with a strong understanding of material characterization techniques are highly sought after in most industrial areas, particularly where material analysis is critical.[6] By establishing a robust foundation in material characterization techniques, students can pursue diverse career paths and significantly contribute to a wide range of research fields.

To overcome the challenges faced by educators, various teaching tools for materials characterization have been developed; these include textbooks, handbooks, and online resources that comprise databases of lecture notes, instructional videos, multimedia platforms, and virtual learning environments.[9−17] Furthermore, subject benchmark statements have been outlined to assist educators in identifying the core concepts within the field.[18] However, while various teaching guidelines have been developed for the broader field of solid-state chemistry, including theoretical frameworks[19−22] and teaching activities based on advanced experimental methods[23,24] and through-research schemes,[25,26] it is worth noting that the development of specific educational frameworks tailored to the domain of materials characterization has been comparatively limited.[27,28]

This study presents a comprehensive teaching and learning framework whose structure is built upon existing frameworks and expands to introduce pedagogical schemes aimed at promoting active learning and developing research skills. The

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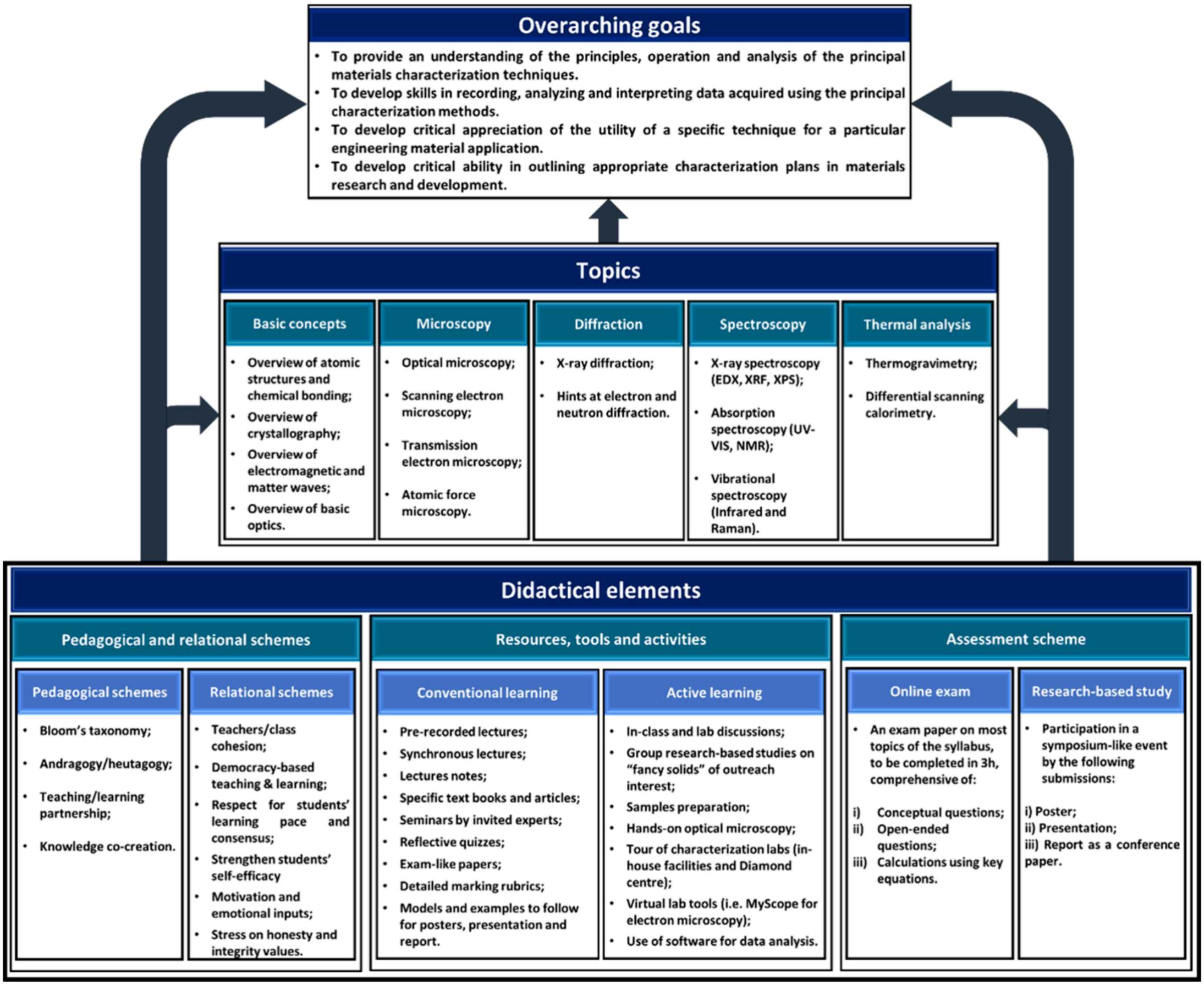

Figure 1. Teaching and learning framework developed for materials characterization.

application of active learning methods is increasing in STEM programs,[29−33] as these approaches tend to engage students in problem-solving, critical thinking, and collaborative learning activities.[33−36] The framework combines asynchronous (pre-recorded) and synchronous (in-person) lectures, as well as research-focused group projects designed to enrich the learning experience. By utilizing group-based laboratory projects followed by presentations, students can develop their teamwork and communication skills. In addition, the framework strengthens the relationship between teachers and students, by fostering a partnership in the learning process. In summary, this study provides a broad and adoptable guide that can be used to design materials characterization modules with a variety of applicable pedagogical tools promoting self-directed and self-determined learning.

THE FRAMEWORK

The framework has been developed for a module titled "Structure and Properties of Materials" delivered to second-year undergraduate students in Materials Science and Sustainable Engineering at the School of Engineering and Materials Science (SEMS) of Queen Mary University of London (QMUL). The module runs for 12 weeks, during which, 18 hours were dedicated to in-person activities. Twelve of these were assigned to synchronous lectures and six to lab activities.

The key components of the framework, the interrelationships, and their mapping are illustrated in the scheme presented in Figure 1. The framework is built upon three main components, identified as

(i) The overarching goals based on the module's learning outcomes.

(ii) The topics specified by the syllabus.

(iii) The instructional components employed in the teaching approach.

The directions of the arrows depict the functionality of each component and the targeted progressions. The framework is of a modular nature, which enables it to be adapted to various contexts.

Overarching Goals

The overarching goals highlighted in Figure 1 aim to provide students with the skills that can be applied for a comprehensive characterization of different materials classes. These include:

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J. Chem. Educ. XXXX, XXX, XXX−XXX

(a) The capability to identify suitable experimental techniques for characterizing the crystal structure and microstructure of materials.
(b) Proficiency in developing appropriate plans for research and development.
(c) A comprehensive understanding of the principles, operation, and analysis of major materials characterization techniques.
(d) Proficiency in collecting, analyzing, and interpreting data obtained through primary characterization techniques.

Topics

The topics taught were aimed at providing comprehensive coverage of the principles, operational procedures, and applications of various characterization techniques, within these four key areas:

(I) Microscopy
(II) Diffraction
(III) Spectroscopy
(IV) Thermal analysis

To facilitate the comprehension of the underlying principles of the primary techniques, the framework is structured into distinct sections:

(1) Basic Concepts: This initial section revisits fundamental knowledge of atomic structures, chemical bonding, crystallography, electromagnetic waves, matter waves, and their interactions with matter.
(2) Microscopy: This part delves into the operational principles, experimental procedures, and real-world applications of optical, scanning electron, transmission electron, and atomic force microscopy.
(3) Diffraction: The main part is dedicated to elucidating the principles of X-ray diffraction, highlighting key characteristics of common diffractometers, and offering insights into the techniques for analyzing and interpreting diffractograms. The main factors affecting the peak's intensity, such as form factor, structure factor, Lorentz factor, multiplicity factor, absorption factor, thermal vibrations, temperature factor, and extinction, are illustrated. The connection between structure factor and electron density, the description of diffraction in the reciprocal space, and the phase problem in crystallography are discussed. Additionally, an overview of electron diffraction techniques and procedures for generating and indexing local diffraction patterns were provided. This section concludes with a survey of neutron diffraction and its advantages over X-ray diffraction.
(4) Spectroscopy: The focus of this part is on various methods and techniques employed for chemical analysis, encompassing energy-dispersive X-ray spectroscopy (EDX), X-ray fluorescence (XRF), and X-ray photoelectron spectroscopy (XPS). This section also covers a few absorption techniques such as ultraviolet and visible light spectroscopy (UV−vis), as well as nuclear magnetic resonance (NMR). The concluding section is dedicated to the principles and applications of infrared (IR) and Raman spectroscopy, contextualized within the vibrational properties of molecules and crystal lattices. The distinction between the mechanisms of interaction between light and matter in both techniques is clarified by illustrating the light absorption process in IR and elastic and inelastic scattering, along with Stokes and anti-Stokes lines and relative Raman shifts in Raman spectroscopy.
(5) Thermal Analysis: This section focuses on the principles and applications of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) as the primary techniques for investigating thermally induced processes and phase transitions.

Didactical Elements

The section of the framework labeled as "Didactical elements" integrates the pedagogical and relational schemes, the teaching resources, tools, and activities, categorized into conventional learning and active learning, as well as the assessment schemes that have been developed.

Pedagogical Schemes. Among the relevant pedagogical schemes, the revised Bloom's taxonomy has been considered to facilitate the attainment of increasingly higher levels of knowledge, through a progression from "remembering" to "understanding", "applying" and "analyzing", followed by "evaluating", and ultimately "creating", with the latter representing the highest level of knowledge.[37] Elements of guided inquiry-based style[25,38,39] have been introduced to realize a combination of andragogy and heutagogy, facilitating self-directed and self-determined learning.[40,41] Through incorporating these techniques, students have been encouraged to seek knowledge based on their individual perceptions, abilities, and preferred resources. Specific instructions and feedback supported students in achieving their objectives and motivated them to set higher targets. Iterative feedback loops, as reported in similar contexts,[27,28] have been included to prevent insufficient or superficial work and misunderstanding of concepts and expectations. Providing students with opportunities to apply feedback enabled quality control, promoted self-directed and self-determined learning, and allowed educators to validate the effectiveness of their feedback and assess the extent of learning.

Relational Schemes. The section on "Relational schemes" includes a series of strategies that have been implemented to foster a positive and collaborative learning environment, influencing students' psychology during the learning process. To facilitate heutagogy, the framework includes diverse elements from various pedagogical strategies reported in the literature as "community and relationship building", "negotiating assessment",[42] and "democracy in learning".[43] Cooperative group activities have been employed to strengthen the relationships among students and between students and educators. One-to-one conversations along with pleasant and respectful gestures have been used to enhance students' comfort in interacting with the educators. By adopting a learner's attitude and perceiving students as peers, educators have been able to establish a sense of shared learning with the cohort. Furthermore, educators have made themselves readily available for clarifications, promptly addressing issues and providing pastoral care when needed. Additionally, respecting the individual learning pace of students has proven crucial for promoting "democracy in learning". Students have been actively involved in moderating the quantity and delivery of the teaching content, as well as arranging deadlines based on their other commitments. This grassroots approach based on students' consensus has improved their motivation, satisfaction, and learning experience.

Drawing on previous research,[44−48] the importance of self-efficacy has been also considered in the present framework. Regular implementation of specialized empowerment strategies

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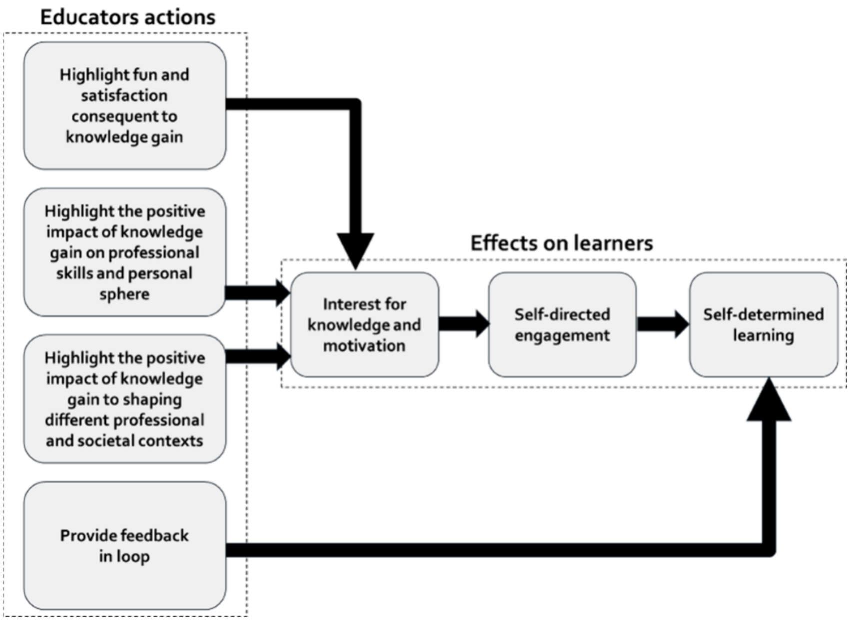

Figure 2. Scheme demonstrating the possible mechanisms to enforce self-determined learning.

to enhance students' self-esteem has taken place during lectures, lab activities, and tutorials, particularly when discussing challenging topics. To enhance self-efficacy, various techniques have been developed to allow students experiencing the four main situations identified by Bandura.[49] Tasks with increasing difficulty allowed students to experience performance accomplishments (Situation 1).[49] Exposure to the work of their peers provided the vicarious experience, demonstrating the attainability of the set targets (Situation 2).[49] Verbal persuasion (Situation 3)[49] has been conducted using specific semantic strategies for encouragement,[50] emphasizing the specific educators' perception of students as individuals progressing toward becoming skilled "scientists and engineers". The stimulation of students' positive emotions (Situation 4)[49] has been attempted through the continuous display of enthusiasm during teaching activities,[51] using praise and rewards, and highlighting the satisfaction that could arise from gaining knowledge. Stress has been mitigated by marginalizing the importance of grades compared to the process of obtaining knowledge and skills. The effectiveness of these strategies was particularly evident when students were repeatedly assured of their ability to deliver presentations without relying on prewritten notes but by actively engaging with the audience. This has proven successful, as none of the students resorted to reading when presenting their case studies. Furthermore, by consistently demonstrating confidence in students' abilities to comprehend and manipulate complex equations, which were intentionally incorporated into the lectures' content, students seemed less intimidated when encountering equations. Figure 2 demonstrates a proposed scheme that can potentially trigger self-determined learning using motivational and emotional approaches, as well as feedback. It has been considered that motivation and interest in knowledge can be increased by

(I) Allowing students to identify the possibility of experiencing pleasure through learning.[52]
(II) Helping students to realize the impact of knowledge on professional and personal spheres.[53]
(III) Teaching students about the necessity of knowledge in shaping societal advancement.

These points are believed to contribute to self-directed engagement and self-determined learning, which can be further driven by the additional support of feedback loops.

The final aspect addressed in the "Relational schemes" in Figure 1 pertains to the promotion of academic integrity, utilizing the ideas presented in the IEPAR framework proposed by Khan et al.,[54] which includes inspiration, education, pedagogical consideration, assessment design, response, and restorative practice. Inspiration has been driven by the educators acting as "role models", demonstrating enthusiasm and knowledge, while maintaining transparency regarding their personal expertise. To further promote integrity, educators have demonstrated good practices, by acting impartially and fairly adopting a supportive approach in the assessment process and emphasizing the importance of fact-seeking during scientific investigations. In addition, students were taught about plagiarism and given detailed instructions regarding referencing procedures. The dishonesty of the practice and its negative effects on undermining individual uniqueness and the ability to provide improvements to existing contexts were highlighted. By combining these pedagogical and psychological prompts, students have been persuaded to not plagiarize.

Resources, Tools, and Activities. This section of the framework includes the didactic materials that have been developed, along with the tools and activities designed to deliver the teaching content. These features are classified into two groups, namely, "Conventional" and "Active" learning tools. The conventional approach incorporates both asynchronous and synchronous lectures integral to the "flipped classroom" model, which has gained widespread popularity in STEM education.[55−58] Based on this scheme, prior to synchronous lectures, students were required to engage in prerecorded sessions, allowing them to actively participate in class discussions. To diversify the teaching delivery and expose students to different teaching styles, experts in specific topics such as NMR and X-ray tomography were invited to deliver a few lectures.

To facilitate learning, detailed and explicit lecture notes were developed for the entire syllabus with the aim of minimizing incompleteness and avoiding dispersion when searching

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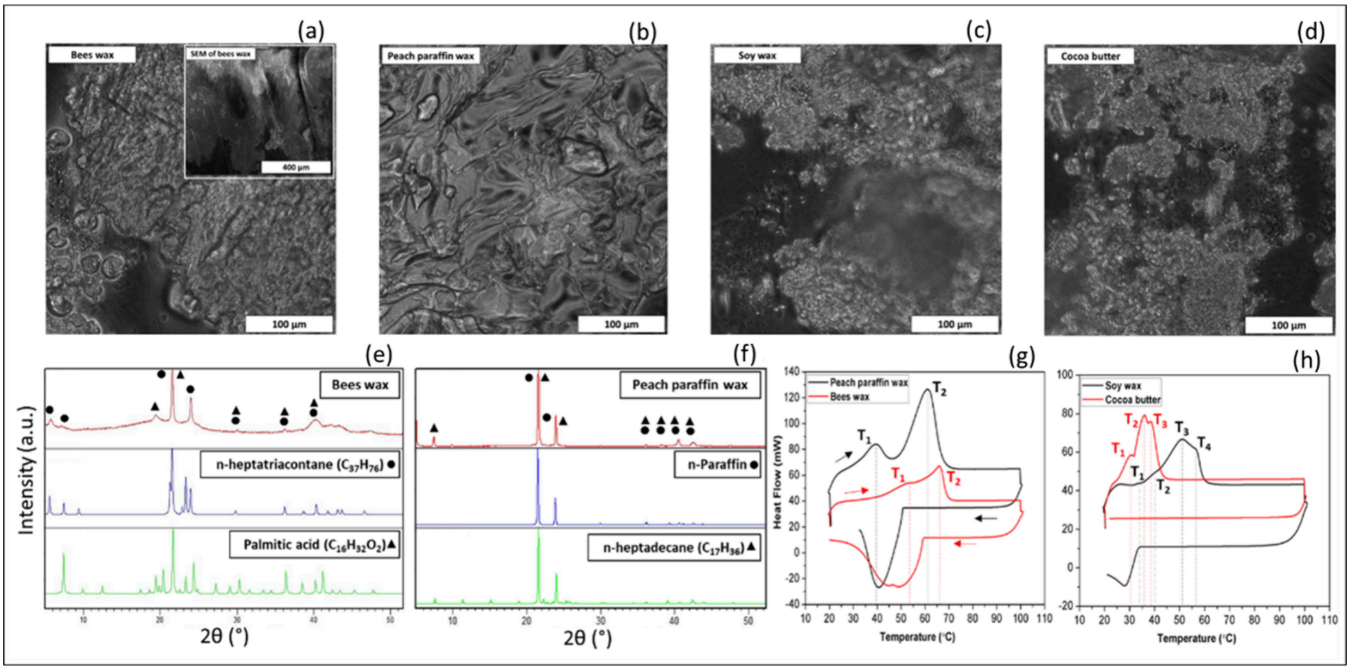

Figure 3. Analysis of waxes. Phase contrast microscopy images of beeswax (a) and SEM image in the inset; phase contrast microscopy images of peach paraffin wax (b); phase contrast microscopy images of soy wax (c); phase contrast microscopy images of cocoa butter (d); X-ray diffraction pattern of bees wax (e); X-ray diffraction pattern of peach paraffin wax (f); differential scanning calorimetry of bees wax and peach paraffin wax (g); differential scanning calorimetry of soy wax and cocoa butter (h).

relevant learning materials. Furthermore, an extensive bibliography of books, articles, and media content was compiled to provide informative and reliable resources. Reflective quizzes have been carefully designed as opportunities for formative assessment to assess students' comprehension, as well as challenge them to apply the acquired knowledge, in accordance with Bloom's taxonomy. These quizzes were based on a combination of "definition quizzes" and "conceptual quizzes" aimed at clarifying and consolidating definition and concepts and "numerical quizzes" designed to perform calculations with relevant equations. Examples of questions included in these quizzes are reported in Section 1 of the Supporting Information. In preparation for the final assessment, a variety of exam-like papers designed for an open-book examination were provided for practice. The questions included contexts that are not readily found in online question databases or previous exam papers. A sample of exam-like papers is included in Section 2 of the Supporting Information.

Active learning involved in-class discussions centered around the topics covered in prerecorded sessions. Additionally, lab tours to in-house experimental facilities were conducted to provide practical learning sessions associated with the lectures' content. In addition, a visit to the Diamond Light Source Centre was also organized to expand the students' perspectives on material characterization.

To increase students' curiosity and engagement, research-based case studies were specifically designed as part of the summative assessments of the module. The case studies focused on classes of materials with a significant outreach interest labeled as "fancy solids". These materials span a wide range of substances and include compounds utilized in diverse sectors such as cosmetics, food, construction, and the electronic industry, as well as materials of particular significance in the field of marine science. To facilitate active learning, students were randomly grouped into teams of three or four members during the second week of the term, and a specific research case was assigned to each group.

The briefings of each case study are reported in Section 3 of the Supporting Information. From the 2nd to the 12th week of the term, students were actively engaged in carrying out research and collecting data relevant to their case study in preparation for a symposium-like event held at the end of the 12th week. The plan for the research activities was outlined in agreement with the students (Gantt Chart in Section 3 of the Supporting Information), supporting the "democracy in learning" process. During the fourth week, students were required to submit their research plans, which underwent a review process. Through the feedback provided, students were guided to integrate microscopy, diffraction, spectroscopy, and thermal analysis techniques for a comprehensive characterization of the materials investigated. The feasibility of the proposed experiments was evaluated. In preparation for electron microscopy and diffraction lab activities, students were asked to use the virtual environment "MyScope"[11] to familiarize themselves with the experimental equipment, procedures, and relevant parameters.

Subsequently, students were engaged in the preparation of the samples for the planned experiments, which were conducted in successive lab sessions. During these sessions, students were provided training and hands-on experience on the actual equipment and were assisted during the planned experiments by Ph.D. students and lab technicians, with the supervision of the lecturers. Optical microscopy required no additional training, as students had already acquired microscope usage skills in previous modules. Conversely, for scanning electron microscopy, students were educated on the main working principles and parameters influencing image quality. During the lab sessions, students were guided through the operational procedures, from instrument settings to image capture. Instead of memorizing practical steps, the training focused on improving image quality and interpretations, covering crucial aspects such

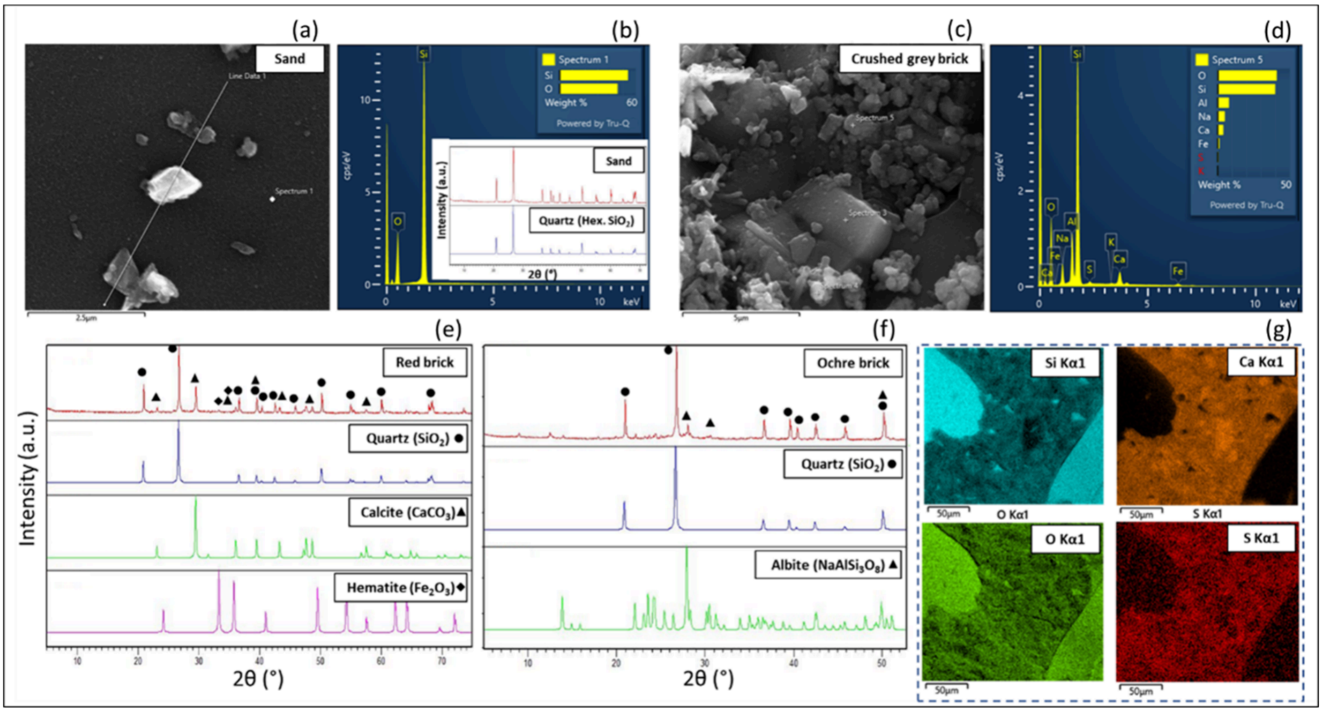

Figure 4. Analysis of construction bricks. EDX analysis (a, b) and X-ray diffraction pattern (inset of b) of sand; SEM micrograph of crushed gray brick (c); EDX point analysis of crushed gray brick (d); X-ray diffraction pattern of a red brick (e); X-ray diffraction pattern of an ochre brick (f); EDX of a reinforced brick (g).

as charging effects, focus, stigmatism, and the respective advantages of secondary electrons and backscattering modes. In the case of EDX spectroscopy, students were trained on the procedures for obtaining chemical compositions through single-point, line scans and area maps. The limitations of EDX techniques in achieving precise chemical analyses were highlighted, while illustrating the benefits of the complementary method, such wavelength dispersive spectroscopy, which could be performed with the same equipment. For X-ray diffraction, an experienced operator demonstrated the technique in the Bragg−Brentano geometry, illustrating the effects of step size and scan time on the diffractograms. Training on X'Pert HighScore software was provided in additional sessions arranged with each group depending on their availability. The straightforward design of the differential scanning calorimetry equipment allowed students to grasp the basic concepts of the experimental procedure while testing their samples. All the trainings undertaken and experiments carried out have helped the students to further understand the working principles and the underlying mechanisms of each technique, consolidating the knowledge acquired from the lectures.

Data collection continued until the eighth week, while the following weeks were dedicated to data analysis using specific software, such as "ImageJ" (U.S. National Institutes of Health) for microscopy and "X'Pert HighScore" (Malvern Panalytical) for diffraction. Additional experiments were conducted in subsequent appointments to fill eventual gaps in the data sets when expressively required by the students.

RESULTS OF RESEARCH-BASED CASE STUDIES

This section showcases the key results obtained from each case study. While some aspects of the present findings require further investigation, it is noteworthy that the quality of the results is comparable with those reported in publications. These outcomes underscore the efficacy of the pedagogical approach outlined in this study for cultivating material characterization expertise among undergraduate students.

Case Study 1: Waxes and Cocoa Butter

Figure 3 includes a collection of results obtained on different types of waxes, with Figure 3a−d showcasing images obtained using phase contrast microscopy (PCM). This technique was chosen by the students as an alternative to scanning electron microscopy and conventional optical microscopy, as the SEM produced beam damage to the samples (see inset of Figure 3a) and the conventional optical microscopes were unable to reveal the microstructure details of the waxes. PCM images show the presence of spherical-like domains in beeswax,[59] soy wax,[60] and cocoa butter[61] and larger microstructural units with more irregular shapes in paraffin wax.[60] Figure 3e,f displays the X-ray diffraction patterns of beeswax and paraffin wax in which the main phases have been identified as *n*-heptatriacontane and palmitic acid in the former and *n*-paraffin and *n*-heptadecane in the latter. Several peaks could not be clearly assigned but could possibly correspond to other less common variants of hydrocarbons and acids[62] that are difficult to unravel at this level of investigation. Figure 3g,h exhibits the thermograms of the waxes, displaying a series of peaks and anomalies. The first peak of paraffin at $T_1 \approx 40$ °C is attributed to a solid−solid phase transformation, while the second peak at $T_2 \approx 62$ °C is associated with the melting point. The peak observed during the cooling process (≈40 °C) is linked to the solidification process.[63] Similar anomalies occurring at higher respective temperatures have been observed in beeswax. Soy wax shows additional anomalies corresponding to various thermally induced transformations.[64] Cocoa butter reveals a series of peaks attributed to the complex polymorphic behavior and melting process.[65] It is worth noting that the thermal analysis data could be used to assess the behavior of paraffin, waxes, and

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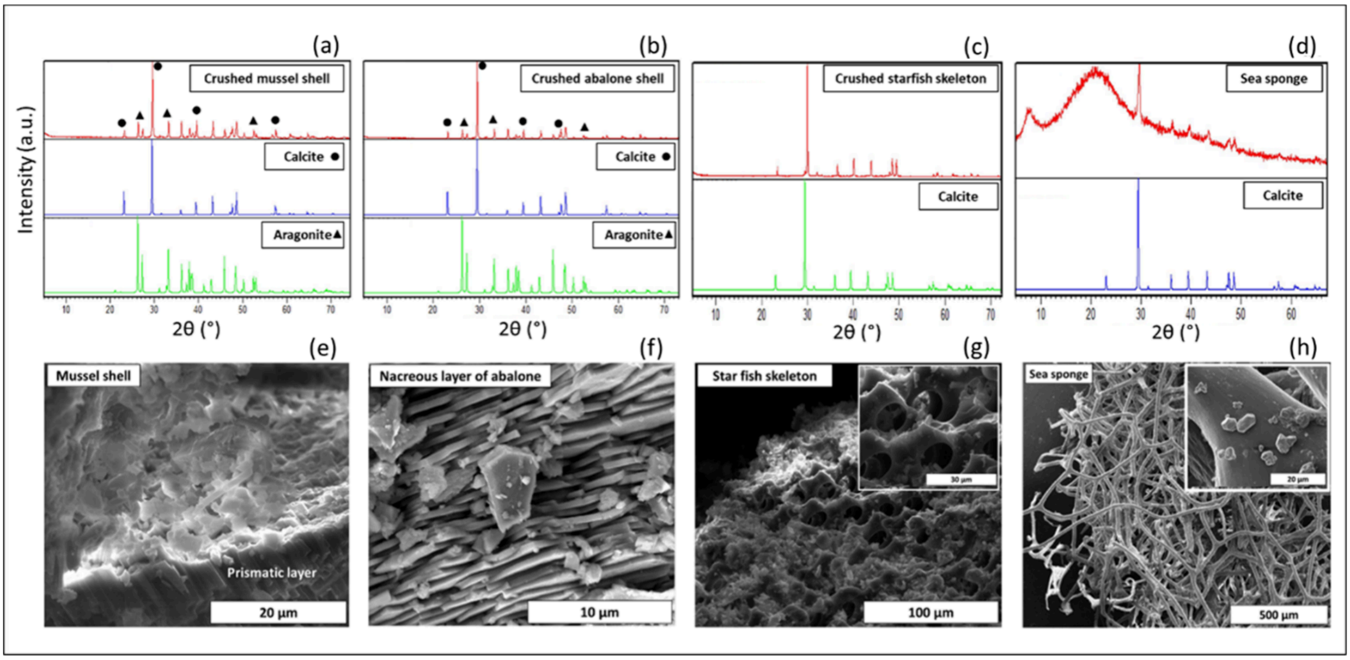

Figure 5. Analysis of marine mollusks. X-ray diffraction pattern of crushed mussel shell (a); X-ray diffraction pattern of crushed abalone shells (b); X-ray diffraction pattern of crushed starfish skeleton (c); X-ray diffraction pattern of sea sponge (d); SEM micrograph of mussel shell (e); SEM micrograph of nacreous layer in abalone shells (f); SEM micrograph of starfish skeleton (g); SEM micrograph of sea sponge (h).

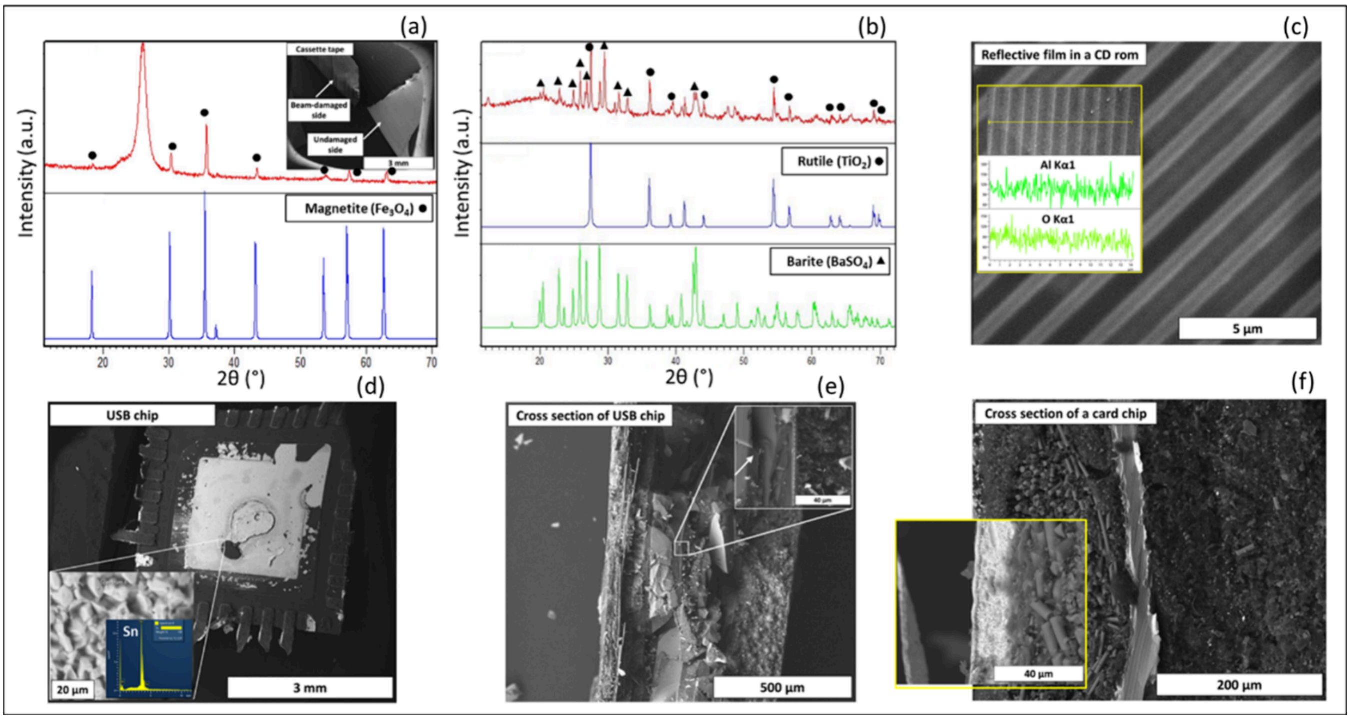

Figure 6. Analysis of memory devices. X-ray diffraction pattern of a cassette tape (a); SEM backscattering image of two sides of a cassette tape (inset of (a)); X-ray diffraction pattern of a reflective layer of a compact disc (b); SEM micrograph and EDX (in the inset) of a reflective layer of a compact disc (c); SEM top view image of a USB chip and EDX in the inset (d); SEM micrograph of the cross-section of the USB chip (e); SEM micrograph of magnetic card chip (f).

cocoa butter as phase change materials and lubricants[63,66−68] and would allow one to identify connections between structure, microstructure, and thermal and tribological properties.

Case Study 2: Construction Materials

Figure 4 displays a selection of data obtained from the analyses carried out on construction materials. Figure 4a−d is relative to a sample of sand and crushed gray brick. The EDX of the former indicates the sole presence of silicon and oxygen, in agreement with the XRD phase analysis that identified quartz as the sole constituent. The SEM of the crushed gray brick shows particles with different morphology and contrast, suggesting the presence of different phases. The EDX analysis on one of the locations

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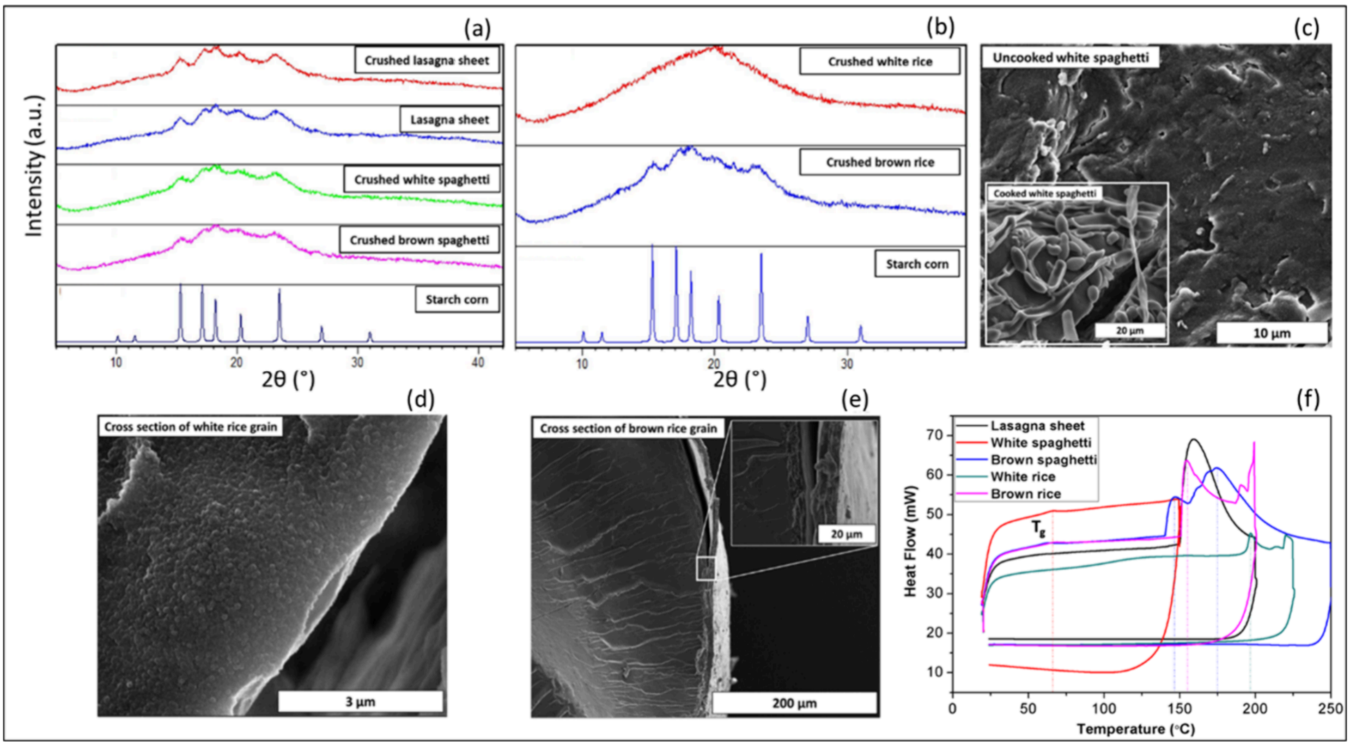

Figure 7. Analysis of pasta and rice. X-ray diffraction patterns of a lasagna sheet and white and brown spaghetti (a); X-ray diffraction pattern of white and brown rice (b); SEM micrographs of uncooked and cooked (inset) spaghetti (c); SEM micrograph of white rice (d); SEM micrograph of brown rice (e); X-ray diffraction pattern of brown rice; differential scanning calorimetry of pasta and rice (f).

indicates the presence of several elements, including silicon (Si), calcium (Ca), aluminum (Al), and iron (Fe), typically found in bricks.[69,70] Figure 4e displays the phase analysis of a crushed red brick that indicates quartz as the predominant phase. Calcite, which should decompose during firing, has also been surprisingly detected. This suggests that the brick has been undercooked or that calcium oxide (CaO) experienced recarbonation.[71] Traces of hematite have also been revealed and are probably responsible for the reddish color.[71]

Various peaks could not be clearly assigned to a specific phase. The phase analysis of the ochre brick in Figure 4f reveals again the presence of quartz as the main phase. Additionally, the possible presence of albite has been detected as reported for other bricks.[71] Also, in the ochre brick, several diffraction peaks remained unidentified. Figure 4g shows the EDX maps generated on a sample of reinforced brick, showing some areas rich in silicon (Si) and oxygen (O), while others rich in calcium (Ca) and sulfur (S). It can be noted that performing mechanical testing on the different types of bricks and relationships between structure, microstructure, and mechanical properties could be established.

Case Study 3: Seashells and Marine Sponge

Figure 5a−d displays the phase analysis conducted by the students on seashells and sea sponges, revealing the coexistence of calcite (main phase) and aragonite in both mussels[72,73] and abalone shells.[74,75] Starfish skeleton only exhibits the presence of calcite,[76] while the sea sponge presents the coexistence of a highly predominant amorphous phase and calcite.[77] The selected SEM images in Figure 5e,f display the typical layered morphology of marine shells, identifying the prismatic layer in mussel shells (Figure 5e)[78] and the nacreous layer in abalone shells (Figure 5f).[75] Students had the possibility of observing the disordered microstructure nearby grain boundaries, highlighting their classification as an areal defect type in polycrystalline solids. Figure 5g highlights the highly porous microstructure of a starfish skeleton, with pores exhibiting a rather regular circular shape and consistent size.[79] Figure 5h shows the entangled fibrous network of the sea sponge and the presence of particles on the fibers' surface magnified in the inset. Insightful experiments to relate microstructure and mechanical behavior might be carried out by atomic force microscopy, which could allow one to estimate mechanical properties in different regions of the seashells' microstructure.

Case Study 4: Electronic Memory Devices

Figure 6 showcases the results of the investigation of materials used in electronic memory devices, revealing the presence of the magnetically active magnetite (Fe_3O_4) in the cassette tape alongside another unidentified phases corresponding to the main diffraction peak (Figure 6a). The contrast in the image generated using backscattered electrons shown in the inset of Figure 6a indicates that the top and bottom layers of the tape are made of different phases;[80] one of these corresponds to the magnetic phase, which experienced beam damage during imaging, as shown in the top-left part of the SEM backscattered image. An interesting investigation relating phase, microstructure, and properties could have been conducted by characterizing the local magnetic hysteresis loops by magnetic force microscopy. Figure 6b shows the diffractogram of the reflective layer of a compact disk that appears unexpectedly complex containing several diffraction peaks that could not be clearly identified. The phase analysis suggests the presence of TiO_2 in rutile form, and probably Barite ($BaSO_4$), although not fully confirmed. Figure 6c shows the presence of regular stripes on the surface of the reflective layer in agreement with a previous study.[81] The EDX in the inset shows the presence of aluminum and oxygen,[82] although no Al-based phases could be clearly

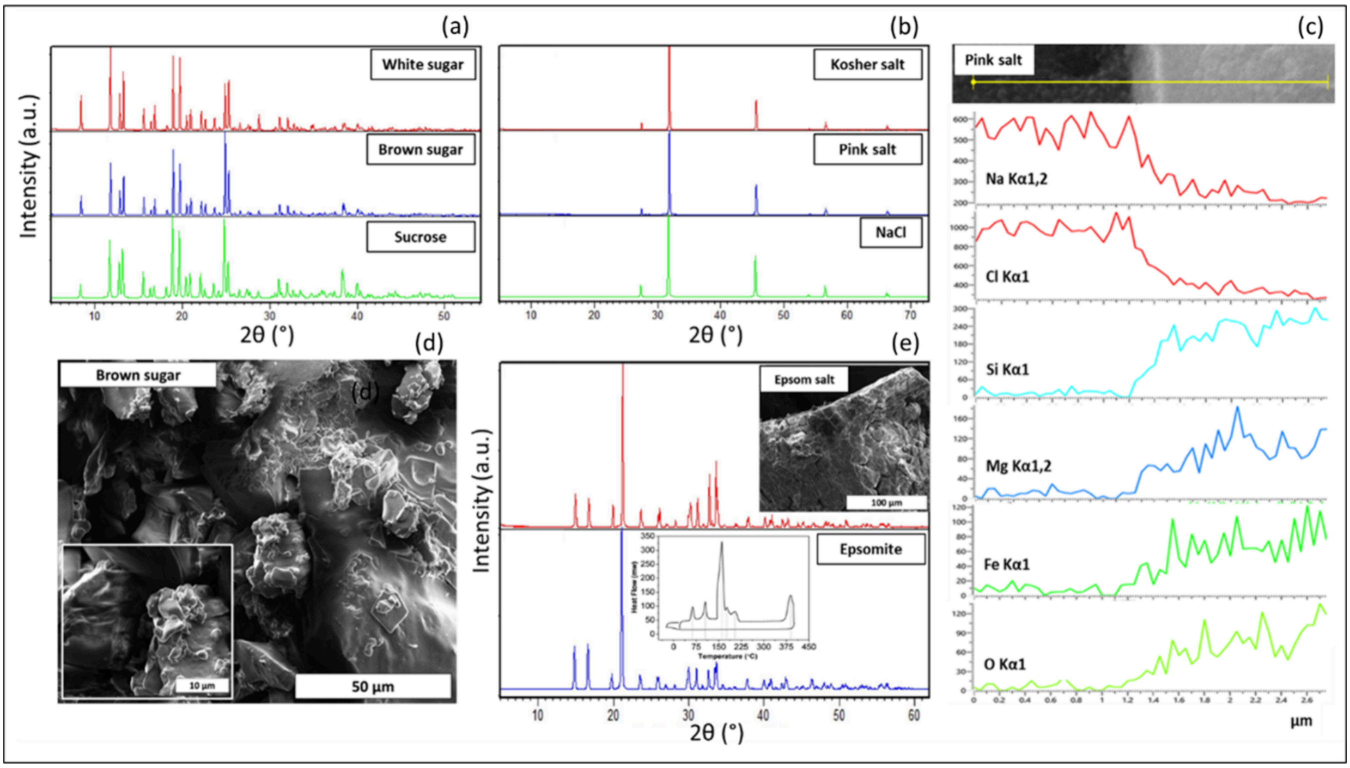

Figure 8. Analysis of salts and sugars. X-ray diffraction pattern of white and brown sugar (a); X-ray diffraction pattern of kosher and pink salt (b); EDX line scan of pink salt (c); SEM micrograph of brown sugar (d); X-ray diffraction pattern of Epsom salt (e) with SEM in the first inset and differential scanning calorimetry in the second inset.

identified in the phase analysis. Figure 6d shows the SEM top-view image of a USB chip, showing the input/output pins placed around the core part. On the chip's surface, there was an obvious presence of tin (Sn), as shown in the inset of Figure 6d, possibly used as a conductive soldering phase. Figure 6e reveals the complex configuration of the USB chip cross-section that contains several layers made of different materials,[83] some of which are separated by regular electrode arrays (follow the white arrow in the inset of Figure 6e). Interestingly, the presence of fibers[84] was observed in the leftmost layer of the image, possibly serving as mechanical reinforcement. The presence of multiple layers made of different materials has been also observed in the cross-section of a card chip (Figure 6f), where the fibrous layer is more clearly visible next to two additional external layers (see inset of Figure 6f).

Case Study 5: Carbohydrates Pasta and Rice

Figure 7 is relative to the types of pasta and rice examined by the students. The X-ray diffraction patterns in Figure 7a,b indicate that the main crystalline phase in these materials is starch,[85−87] alongside an amorphous phase, which appears to be the main constituent of white rice. However, rice's diffractogram does not show distinct peaks. As for the pasta, the comparison between crushed and as-received lasagna sheets suggests the absence of texture in the latter. Figure 7c shows the SEM micrographs of uncooked white spaghetti and the formation of a fibrous-like phase during the cooking process (inset of Figure 7c).[88] Figure 7d,e compares the cross sections of white and brown rice grains, with the former exhibiting spherical-like grains. In the latter, an external layered shell encasing the core part can be observed. Figure 7f shows the thermograms of pasta and rice types, highlighting a small bump at about T_g = 65 °C in white and brown spaghetti as well as brown rice. This can be attributed to the so-called gelatinization temperature.[89] The peaks at higher temperatures correspond to the thermal decomposition process of starch and to other possible irreversible thermally driven processes that occur during heating. By performing rheological tests at different temperatures, the thermal behavior could be related to the flow properties of pasta types, which might be relevant for the cooking and industrial processing of spaghetti and lasagna types.

Case Study 6: Crystal Salts and Sugars

Figure 8 is relative to the sugar and salt crystals. Using the XRD patterns in Figure 8a, it was found that both white and brown sugar crystals are made of monoclinic sucrose. Cubic rock salt appears to be the main phase in kosher and pink salt, with the latter showing additional small peaks corresponding to impurities (Figure 8b). These impurities contain additional elements, such as silicon (Si), magnesium (Mg), and iron (Fe) shown in the EDX line scan in Figure 8c that may be responsible for the perceived pink color.[90] Figure 8d shows the SEM micrograph of brown sugar highlighting particles of different shapes and morphology, probably due to the presence of molasses. Figure 8e displays the diffractogram of Epsom salt. Its phase was identified as epsomite $MgSO_4 \cdot 7H_2O$, which undergoes a dehydration process characterized by different stages as shown in the DSC thermogram reported in the inset.[91] The microstructure shown in the inset of Figure 8e indicates the presence of cracks and streaks on the surface of the crystals.[92]

■ THE SYMPOSIUM

One of the most important elements of the assessment approach implemented in this module is the symposium event. It was created to serve as the culminating activity after the case studies, purposefully called "Structure and Properties of Fancy Solids, 1st

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J. Chem. Educ. XXXX, XXX, XXX−XXX

Edition", with the aim of establishing a recurring tradition for future years, accumulating knowledge generated by students on this category of solids. The event was promoted on Eventbrite, and a specific logo and brochure (Section 5 in the Supporting Information) were created to encourage guest attendance. The invitations were extended to Ph.D. students, researchers, and academic staff of the school, emphasizing the significance of the event.

The objective of the symposium was to provide students with an immersive experience resembling a specialized conference on materials science, offering them an opportunity to showcase their data and interact with peers in the field. Each group was required to submit a poster and the slides used for their presentations prior to the symposium, where the posters were displayed. The duration of each presentation was set to 15 min, allowing every group member to present their part. The Q&A sessions for each presentation were open to all attendees. Following their presentations, students positioned themselves near their posters for further discussion. In preparation for the event, model examples of posters and presentations were made available to the students. For the presentations, they were advised to emulate the performance of speakers presenting their research at events such as the "Young Persons' Lecture Competition" organized by the Institute of Materials, Minerals and Mining and to adopt styles from peer-reviewed journals such as the "Journal of Visualized Experiments" and "Science Talks". While students were free to choose the style and layout for their presentations, the main requirement was that the talks should be delivered without reading notes. They were trained in presentation skills from Week 9 and were guided on confidence-building techniques.

To allow students to incorporate eventual insights obtained during the symposium discussions into their reports, the deadline for submitting the first draft was set 2 days after the symposium. The submissions were then reviewed, and the students had the opportunity to improve their reports based on the feedback provided. They were then given 2 weeks to submit their revised versions as per their preference. As reported by a pedagogical approach used in a similar context,[36] the feedback implementation allowed students to become familiar with the peer-review and publication process of research papers.

■ ASSESSMENT

The assessment included a summative exam and an evaluation of the posters, presentations, and reports. The exam paper constituted 50% of the module marks and contained four main questions, with subquestions that aimed to cover most of the module topics. They included conceptual inquiries as well as open-ended questions involving plot analysis and calculations. The exam was conducted online, within a 3 h window and 2 h for completion. The number of questions and the duration were carefully designed to prevent collusion. To support the efficacy of the framework toward understanding of the underlying principles and mechanisms of the characterization methods considered, the exam paper of the first sit and the relative marks distribution for each question are reported in Section 4 of the Supporting Information. In particular, Figure S3 plots the percentage of the maximum grade assigned to each question against the percentage of students scoring such grades. The remaining 50% of the module's marks was allocated to the research case study, divided between the poster, the presentation, and the final report. The marking criteria for each component were clearly communicated to the students along with the assessment instructions at the beginning of the semester.

■ MODULE EVALUATION AND STUDENTS' FEEDBACK

The students' reception of the framework's implementation was assessed through an internal questionnaire. The results indicate a high level of satisfaction among the students in several aspects of the module, and the feedback provides evidence of the module's success in fulfilling the intended objectives and providing a valuable learning experience. The outcome is relative to six key questions presented in Figure 9. The chart indicates the percentage of students giving a score in the range of "0−5" for each of the following questions.

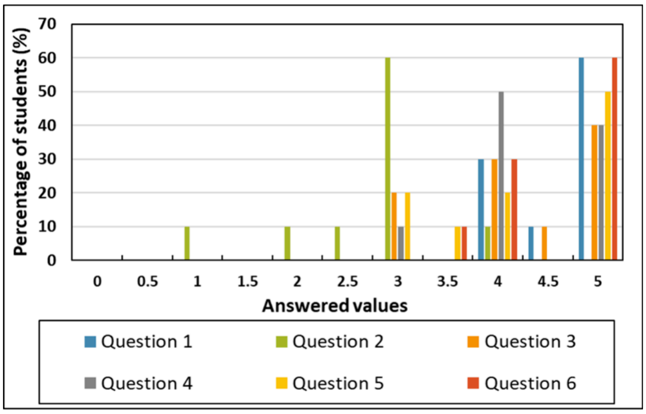

Figure 9. Students' responses to the following feedback questions. Question 1: Rate the module content using a score in the range of 0−5, with 0 rating it very unsatisfactory and 5 very satisfactory. Question 2: Rate the difficulty of the module's content using a score in the range of 0−5, with 0 rating it very difficult and 5 very easy. Question 3: Rate the benefit of the topics covered in this module for your program of study using a score in the range of 0−5, with 0 rating it poorly beneficial and 5 highly beneficial. Question 4: Rate the quality of the resources provided to prepare for the coursework assessments using a score in the range of 0−5, with 0 rating it not beneficial at all and 5 very beneficial. Question 5: Rate your laboratory experience with a score in the range of 0−5, with 0 rating it very poor and 5 very good. Question 6: Rate the educational level of your laboratory experience using a score in the range of 0−5, with 0 rating it not educational at all and 5 highly educational.

(I) The perception of the module (Question 1) received positive feedback, with all students giving scores of 4 and above.
(II) The level of difficulty (Question 2) was perceived as relatively high, as evidenced by the average score and the variability in the students' responses, indicating that the module presented challenges and required significant effort from the students. This outcome can be perceived as beneficial for learning and personal growth.
(III) The usefulness of the topics covered in relation to the program of study (Question 3) was rated positively, with 70% of the students giving scores of 4 and above.
(IV) The available resources (Question 4) have also been positively judged, with more than 90% of the students rating them with scores of 4 and above.
(V) The success of the lab-based sessions (Question 5) received high ratings, with 80% of the students giving scores of 4 and above.

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J. Chem. Educ. XXXX, XXX, XXX−XXX

(VI) The educational value of the lab activities (Question 6) was highly appreciated by the students, with 90% giving scores equal to or higher than 4. This suggests that the hands-on practical sessions provided valuable experiential learning opportunities and enhanced the students' understanding and application of the module's concepts.

CONCLUSION

This work presents a comprehensive teaching and learning framework for the discipline of "Materials Characterization". The framework combines a diverse range of pedagogical resources, strategies, and innovative ideas specifically designed to enhance student engagement and research skills. Key elements include active learning and relational approaches that facilitate self-directed and self-determined learning, as well as a learning partnership between educators and students. The research case studies have served as an immersive hands-on experience in materials characterization methods that could not be realized using other means such as images, illustrations, demonstrative videos, and other ex situ tools. The activities carried out have not only consolidated the understanding of characterization techniques but also enhanced the students' research skills and generated outreach knowledge in various categories of materials. The positive feedback received from students confirms the significant impact of the framework on their understanding of materials characterization techniques and the overall learning experience. Educators in similar contexts can adapt and further refine this framework to promote an effective learning experience in materials characterization, exposing students to open-ended research problems and preparing them for successful careers in the fields of science and engineering.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.3c00974.

Section 1: Examples of questions included on quizzes for formative assessment; Section 2: A sample of exam-like papers; Section 3: Description of case studies and relative plan; Section 4: Exam paper for the first sit and brief discussion on the relative marks distribution (PDF; DOCX)

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Notes

The authors declare no competing financial interest.

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