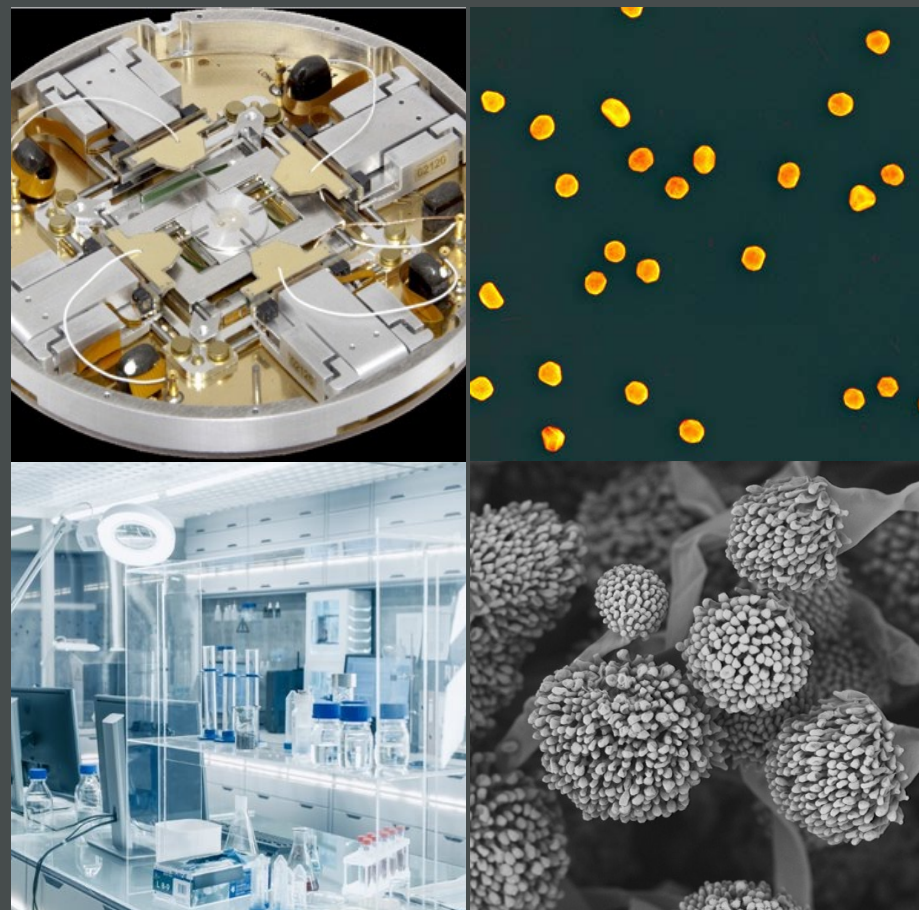


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## A Brief Guide to Characterization of Nanostructured Materials

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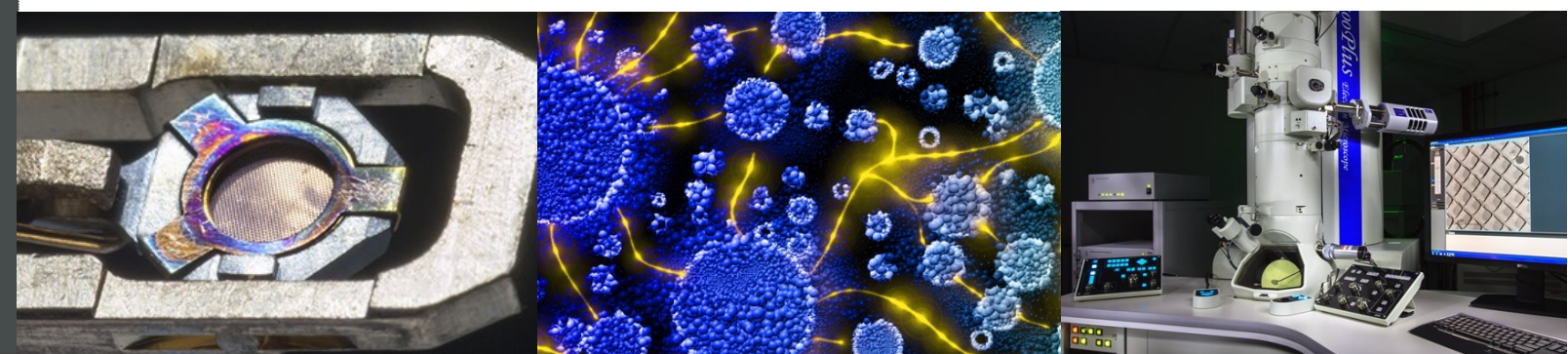
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## Contents

Characterization of Nanostructured Materials Research Overview	1
Importance of Characterization of Nanostructured Materials?	2
Current research in Characterization of Nanostructured Materials at University College Dublin and how this could impact industry?	3
Application of research into Characterization of Nanostructured Materials	4
Summary	5



## Characterization of Nanostructured Materials Research

Nanostructured materials are one the most rapidly growing class of materials due to their unique features such as size, shape, optical and electronic properties, mechanical strength, chemical activity that are profoundly distinct from their bulk analogues. The unique features of those materials, such as high reactivity and surface area, exceptional strength, biodegradability (in some cases), easy chemical functionalization and etc makes those materials suitable for many applications in healthcare (targeted drug delivery and tissue engineering), electronics (due to their special electronic properties they are used to manufacture storage devices), textile Industry (manufacturing of functional fabrics, with exceptional strength or hydrophobicity) and environmental industry (purification using the high reactivity due to high surface area and porosity). These features make those materials highly attractive but at the same time lead to a lot of challenges and difficulties when it comes to their characterization, which is the in depth study of individual features of nanostructured materials and is essential to understand and control their behaviour. The nanotechnology field encompasses a wide array of areas including nanomedicine, nanolithography, tissue engineering etc each of which have distinct needs in terms of characterization techniques and methods. Indeed, the existing characterization techniques have been developed to serve the needs of these different areas, such as the required accuracy and resolution criteria, and often researchers and specialists from different fields are involved in the development of a new method or optimisation of one that is currently used.

A key group of characterization techniques is microscopy based and include both Electron Microscopy (scanning and transmission electron microscopy) and High-resolution Optical Microscopy which provide information about the nanomaterial size, morphology, chemical composition, surface chemistry, optical properties, and crystal structure. Electron microscopy was first developed a few decades ago and significant advancements were made over the years to improve the resolution (up to atomic level), sample preparation and preservation during observation (cryogenic conditions essential for biological samples). One of the biggest advances in the field was the development of Correlative Light Electron Microscopy (CLEM) that is used to study nanoparticles - cell interactions at a very high resolution, providing important information on cellular trafficking and uptake. Electron as well as optical microscopy is often used in conjunction with other characterization techniques including the size measurement techniques based on scattering, surface chemistry measured by infrared spectroscopy, elemental composition by mass spectrometry, and crystal structure by X-ray diffraction. A lot of those techniques have been developed in other scientific fields and then applied to characterization of nanomaterials. Therefore, special attention is required when choosing suitable controls, performing calibration or acquiring the amount of sample required. The main drawback of using advanced microscopy methods is that they are still very expensive, hard to maintain, do not give good statistical information and introduce a lot of artifacts in the sample. Nevertheless, they enable us to observe the nanosized objects in their closest to real state shape and morphology.

## Importance of Characterization of Nanostructured Materials?

Prior to the use of any nanostructured materials in research studies or industrial process, an in-depth characterization involving wide range of instrumentation is required to make sure that the materials meet certain quality and purity criteria, and they carry their application defining properties. The type of analysis and characterization required is defined by the application of the material. For example, researchers may want to characterize specific properties like size and shape, during and after synthesis to evaluate process modifications, or to estimate endotoxin contamination if the material will be used for “in vivo” and “in vitro” studies where inflammatory reaction can be a problem. For manufacturers that use nanomaterials as a part of their production, it is crucial to verify that feedstock they are using from a supplier meets certain criteria (purity, surface area, chemical composition, size, porosity etc) to ensure their final product exhibits the required properties. When it comes to the safety and use of nanomaterials in a work environment, a very important aspect in characterization is their toxicity, which is evaluated using special protocols that require information about purity, crystallinity, surface area, or other properties that are known to have biological relevance.

Current nanomaterial characterization approaches and methods have their own strengths and limitations and often must be used in conjunction to get a full and accurate picture. For the majority of nanostructured materials, a combination of various techniques is required for their in-depth characterization in terms of size, shape, surface components and chemical composition. While these methods are highly effective in research laboratories to understand and control materials, it may not always be possible to apply them at the industrial scale during the bulk production of materials as they require high expertise and relatively long analysis time, and they can be costly. As such, academia-industry collaborations are often established with the aim of better understanding products and processes.



## Current research in Characterization of Nanostructured Materials at University College Dublin and how this could impact industry?

The Centre for BioNano Interactions at University College Dublin focuses on advanced quantitative bio-nanoscience and have unique expertise in understanding the behaviour of nanoparticles in biological environments. To do so the researchers of the Centre have established specialised facilities and platforms to carefully analyse objects at the nanoscale. These include a specified facility for nanomaterials synthesis and characterization in a “clean” environment, that is required to prepare nanomaterials for use in vivo and in vitro experiments. The nanomaterial characterization platform at CBNI enables an in-depth investigation of a range of nanomaterial features using state-of-the-art equipment & protocols, and the Centre also has access to the imaging facility in the campus. With its current platform and experience the researchers at CBNI are able to meet the needs of the industry by offering optimisation of their current characterization protocols, by providing in-depth information about their products using advanced methods that are not widely used in the industrial sector, or by developing a completely new workflow for characterization and quality control.

## Application of research into Characterization of Nanostructured Materials?

Nanotechnology finds various applications in life sciences in areas like therapeutics, diagnostics, tissue engineering and drug delivery. To design and engineer nanomaterials for such applications, nanomaterials need to be observed and studied in a state as close as possible to the real environments they will be in (e.g. in blood or organs) where they will interact with living matter and objects (e.g. human cells). Indeed, the goal of advanced characterization and analysis methods is to study nanomaterials in laboratory conditions that mimic real-life environments.

One of the biggest advancements has been the development of electron microscopies working at very low temperatures that helps prevent structural disintegration. These techniques known as Cryo-TEM and Cryo-SEM, allow the analysis of biomolecular structures (e.g. proteins) as they would exist in a physiological conditions (e.g. in the blood) and at near-atomic resolution which is crucial to develop certain applications using nanomaterials such as drug delivery. Another recently developed technique that could be used for nanomaterial characterization when interacting with biological objects is the Correlative Light and Electron Microscopy (CLEM). With this technique the cellular and nanoparticles structure can be revealed at high resolution and at the same time their localization within living cells can be detected. This technique is expected to bring a lot of new insights in the field where the interaction between the nanomaterials and biological objects can be observed in great detail and in realistic conditions.

## Summary

The development and availability of techniques that allow physical, chemical, and biological characterization of nanomaterials are essential to ensure the further growth of application areas, manufacturing and commercialisation. A wide variety of analytical detection tools and characterization approaches are the basis to understanding how nanomaterials behave under different conditions, like the complex manufacturing processes they undergo or in research studies where they interact with the biological systems. For safe and efficient application of nanostructured materials in industry, access to reliable and comprehensive set of characterization tools, providing informative and reproducible data is essential. The unique properties of those materials are defined by the broad range of parameters (e.g. size, shape, particle size distribution and stability, surface area, surface composition), which must be carefully monitored as even small variations may lead to the loss of an essential characteristic. To expand the use of these materials and look for new applications in the industry, the manufacturers need to have a deep understanding of these characteristics. The development of time and cost-efficient characterization protocols and quality control measurements are prerequisite for their final product development and innovation. Another important step is the careful inspection of any raw nanomaterial used in the manufacturing process to assure the quality of the final product, verify potential batch to batch variability and gain awareness of potential harmful and toxic effects those materials could have to the environment or the humans exposed to them.



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