

Chapter 3

Analytical Techniques for Characterization of Nanomaterials

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ABSTRACT

With the increasing demand of nanoparticles in various applications, many challenges are also noticed in terms of synthesizing accurately designed nanomaterials. The characteristics to be customized in order to make these nanomaterials useful need characterization and verification using different tools that have certain limitations and significant strengths. The purpose of this chapter is to provide the description of measurement methods that may be applied to nanodimension in order to study their most important physical and chemical features. This chapter will provide a detailed introduction, mechanism of working, and limitations of various characterization tools. Focus will be on giving detail about widely applicable and commonly approachable methods to measure properties that can be categorized in terms of size, size distribution, aggregation, shape, dimension, surface area, composition, surface chemistry, charge, surface potential, and functionally active interactive groups.

INTRODUCTION

There is a significant difference between the physical or chemical properties exhibited by nanoscale materials and bulk materials of the same composition. This is because of obvious variation in surface to volume ratio, surface plasmon resonance, reactivity and electronic interactions. This is why nanotechnology and nanomaterials have attracted researchers around the globe. Nanomaterials are synthesized using different conventional and unique methods which can be mechanical, ionic, biologically assisted, laser induced or chemical in their nature. Miniaturization to nanoscale is crucial in order to successfully

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conduct experiment without wasting up the resources and using the results for improving the quality of life (Koczur, Mourdikoudis, Polavarapu, & Skrabalak, 2015).

“Nano” means “a billionth,” i.e., in nanotechnology is the study of materials having dimensions of the order of a billionth of a meter. It is actually not clear when humans first began to take advantage of nanosized materials. Back in fourth century, Roman glass makers reportedly fabricated color glass containing nanosized materials as Lycurgus glass which contained silver and gold nanoparticles. Today, nanotechnology is a set of advance scientific methodologies to manipulate the material at nanoscale. This fastest growing area of science and technology is of emerging importance so much that investment in nanotechnology research is reported to increase many folds in fact reaching millions of dollars since it started. On the broader scale, usage/ fabrication/ exploration of objects in nanometer dimensions in order to reveal their applications in chemical, physical or biological worlds comes under the sky of nanotechnology.

Nanoparticles play significant role as medication when their role in biomedical applications is considered. Moreover, the high surface area to volume ratio enhanced colloidal stability make them most attractive for biomedical and sensing applications. In addition, size restriction of materials, imparts absolutely different properties to nanoparticles as compared to their bulk counterparts. For instance, Surface plasmon resonance (SPR) band as exhibited by some metal nanoparticles is not observed in bulk metal. This phenomenon is highly affected by the size, shape, surface chemistry, core electrostatic properties and surrounding temperature. Currently, great innovative products are being synthesized using nanotechnology which is further used in medical, industrial, agricultural and even crime investigation fields. This large number of applications and synthesis call for precise, authentic and up-to-date characterization.

Nanometrology is the science of measuring at nanoscale which is not only limited to measurement of size or length of nanoparticles/ nanomaterials. It also deals with analysis of chemical composition, optical properties, mass, electrical and other physical or chemical properties. There are many nanometrological techniques used for instance SEM, TEM, AFM, XRD, and EDX which are used to obtain morphological data about nanoparticles and various other studies are performed to analyze electrical behavior of nanoparticles. However, the study of nanomaterials using these techniques comes with its own challenges where inherent properties of nanomaterials may hinder accurate study of their features, insufficient production at laboratory scale and the multidisciplinary nature of this research also affect access to characterization tools required for specific purposes. It is, therefore, necessary to study the usability of data obtained from characterization tools, limitations of these technique and strengths of the tool keeping the application in mind (Wang et al., 2013).

Different techniques of characterization can be categorically grouped according to their usage. For example, microscopy-based methods for surface, morphology, size data (TEM, FESEM, AFM, etc.), magnetic techniques to study certain type of groups (SQUID, VSM etc.) and techniques for structural or elemental properties (X-ray spectroscopy, XRD, UV-Vis spectroscopic techniques). Table 1 will summarize some of the important parameters and characterizations tools to measure them.

BACKGROUND

Nanotechnology is focused on using the advantage of unique characteristics of materials at nanoscale. With its vast usability in health, energy, forensic, agriculture, consumables and other industries, it has directed research towards itself from around the globe. Nanomaterials are not defined in terms of a specific

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