



## Review

# Instrumental methods and techniques for structural and physicochemical characterization of biomaterials and bone tissue: A review



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## ABSTRACT

A review of recent advances in instrumental methods and techniques for structural and physicochemical characterization of biomaterials and bone tissue is presented in this paper. In recent years, biomaterials attracted great attention primarily because of the wide range of biomedical applications. This paper focuses on the practical aspects of instrumental methods and techniques that were most often applied (X-ray methods, vibrational spectroscopy (IR and Raman), magnetic-resonance spectroscopy (NMR and ESR), mass spectrometry (MS), atomic absorption spectrometry (AAS) and inductively coupled plasma-atomic emission spectrometry (ICP-AES), thermogravimetry (TG), differential thermal analysis (DTA) and differential scanning calorimetry (DSC), scanning electron microscopy (SEM), transmission electron microscopy (TEM)) in the structural investigation and physicochemical characterization of biomaterials and bone tissue. The application of some other physicochemical methods was also discussed. Hands-on information is provided about these valuable research tools, emphasizing practical aspects such as typical measurement conditions, their limitations and advantages, interpretation of results and practical applications.

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**Abbreviations:** A, Androstane; AAS, Atomic absorption spectrometry; AFM, Atomic force microscopy; AL, Alginate; BCC, Biphasic calcium composite material; BCP, Biphasic calcium phosphate; BHAP, Biological hydroxyapatite; BPs, Bisphosphonates; CA, Contact angle analysis; CDHAP, Calcium-deficient hydroxyapatite; Ch, Chitosan; CHAP, Carbonated hydroxyapatite; CoHAP, Cobalt-exchanged hydroxyapatite; CP, Calcium phosphate; CP/PLGA, Calcium phosphate/poly-D,L-lactide-co-glycolide; DLPLG, Poly-D,L-lactide-co-glycolide; DMEM, Dulbecco's Modified Eagle's Medium; DSC, Differential scanning calorimetry; DTA, Differential thermal analysis; EDX, Energy dispersive X-ray spectroscopy; EELS, Electron energy-loss spectroscopy; EFM, Electrostatic force microscopy; ESCA, Electron spectroscopy for chemical analysis; ESR, Electron spin resonance; FAP-TiO<sub>2</sub>, TiO<sub>2</sub> coated with fluoridated apatite; FE-SEM, Field-emission scanning electron microscopy; FTIR, Fourier transform infrared spectroscopy; Gel, Gelatin; GC, Gas chromatography; GPC, Gel permeation chromatography; HAP, Hydroxyapatite; HAP/PLLA, Hydroxyapatite/poly-L-lactide; HPLC, High performance liquid chromatography; HRTEM, High-resolution transmission electron microscopy; ICP-AES, Inductively coupled plasma-atomic emission spectrometry; ICP-OES, Inductively coupled plasma optical atomic emission spectrometry; KET-NP, Ketoprofen-loaded nanoparticles; LC, Liquid chromatography; MALDI-TOF MS, Matrix-assisted laser desorption-ionization time of flight mass spectrometry; MAS, Magic-angle spinning; MFM, Magnetic force microscopy; MS, Mass spectrometry; Mt, Montmorillonite; NIR, Near infrared spectroscopy; NMR, Nuclear magnetic resonance; NP, Nanoparticle; PCS, Photon correlation spectroscopy; PDLLA, Poly-D,L-lactide; PLGA, Poly-D,L-lactide-co-glycolide; PLGA-PEG, Poly(D,L-lactide-co-glycolide)-poly(ethylene glycol); PSC, Partial stabilized cements; PSD, Particle size distribution; PVA, Polyvinyl alcohol; ROS, Reactive oxygen species; SAED, Selected area electron diffraction; SBF, Synthetic body fluid; SCM, Scanning capacitance microscopy; SEM, Scanning electron microscopy; SERS, Surface-enhanced Raman spectroscopy; Si-Ap, Silicon substituted apatite; SIMS, Secondary ion mass spectrometry; Si-TCP, Silicon stabilized tricalcium phosphate; SPM, Scanning probe microscopy; ssNMR, Solid-state nuclear magnetic resonance; SThM, Scanning thermal microscopy; STM, Scanning tunneling microscopy; TCP, Tricalcium phosphate; TDM, Therapeutic drug monitoring; TEM, Transmission electron microscopy; TG, Thermogravimetry; TOF MS/UPLC, Time of flight mass spectrometry/ultra-performance liquid chromatography; TTCP, Tetracalcium phosphate; UHMWPE, Ultrahigh molecular weight polyethylene; WAXS, Wide angle X-ray structural analysis; WDX, Wavelength-dispersive X-ray analysis; WMTA, white mineral trioxide aggregate; XPS, X-ray photoelectron spectroscopy; ZOL, Zoledronic acid; ZP, Zeta potential.

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## 1. Introduction

Biomaterials include a broad class of materials that are used in medicine and dentistry such as: metallic biomaterials, ceramic and glass biomaterials, non-degradable synthetic polymers, biodegradable polymers, bio-derivative polymers as well as passive and bioactive coatings [1,2]. The biocompatibility, mechanical properties, absence of toxicity of biomaterials or their degradation products are the most important characteristics for biomaterials applications. Biomaterial characterization is based on the combination of theoretical and experimental methods and successive experimental testing, which indirectly leads to the spread of relevant scientific knowledge through the establishment of universally accepted model phenomena in particular materials, which then enables their practical applications. Existing experimental instrumental techniques for characterization of biomaterials could be divided into methods for characterization of spatial structure, measurement of surface characteristics, and determination of the composition and structure of biomaterials, phase transformations and molecular weight

distribution [3]. Structural characterization of biomaterials is of great importance in preparation of scaffolds that are widely used in tissue engineering [1–3]. To date, the field of tissue engineering has the great success, especially in the area of bone tissue, where tissue-engineered constructs have been used successfully in patients. The research is focused on the development of materials with, for example, bio-instructive and stimuli-responsive properties [4]. In this paper, instrumental methods and techniques that can be used for characterization of different biomaterials and bone tissue are summarized (Fig. 1).

Over past years, significant progress has been made in the development of biodegradable polymeric materials. The biomedical applications of these materials, demands specific physical, chemical, biological, biomechanical and degradation properties [5].

A comprehensive systematic review which includes a large number of methods that can be applied for the characterization of biomaterials and bone tissue widely used in biomedical applications could be very valuable for gaining better insight into the advantages and shortcomings of particular techniques and usefulness of the obtained results.

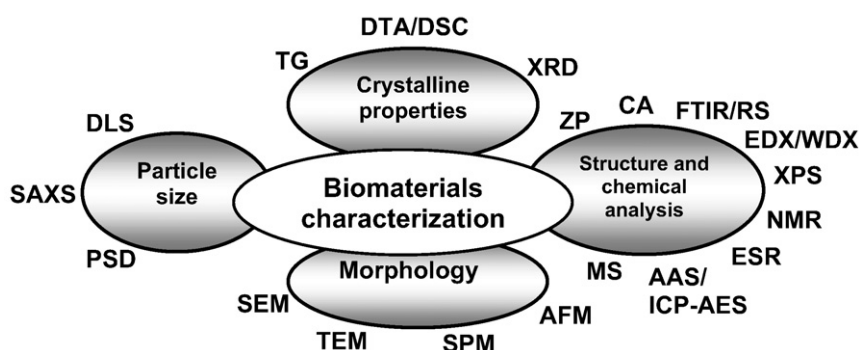


Fig. 1. Various techniques for biomaterials characterization.

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