Detailed Study Notes: Physical and Chemical Characterization of Biomateria

These detailed study notes are based on the chapter "Physical and Chemical Characterization of Biomaterials."

They elaborate all the concepts, techniques, and applications with explanations and examples for exam preparation.

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1. MICROSTRUCTURAL CHARACTERIZATION

- Properties of biomaterials are influenced by crystallography (atomic arrangement), morphology (size, shape, distribution), and composition (chemical makeup).
- Tools: Optical Microscopy, SEM, TEM.

OPTICAL MICROSCOPY:

- Simplest form of microscopy using visible light (400-700 nm).
- Resolution limited to ~200 nm due to diffraction limit.
- Magnification up to 1000x.
- Contrast modes: Bright field, Phase contrast, Fluorescence, Confocal microscopy.
- Example: Titanium alloys (cpTi) studied for grain refinement. Grain size reduction by Severe Plastic Deformation increases strength and bioactivity.

TRANSMISSION ELECTRON MICROSCOPY (TEM):

- Uses electron beams (wavelength << light), resolution <1 nm, magnification up to 2 million.

- Modes:

  * Bright-field: Contrast from thickness, atomic number, diffraction.

  * Dark-field: Uses diffracted beams, highlights defects and crystallinity.

  * High-resolution TEM (HRTEM): Lattice images at atomic resolution.

- SAED (Selected Area Electron Diffraction) patterns used for identifying single crystal, polycrystalline, or amorphous nature.

- Example: NiTi (Nitinol) alloy studied for Shape Memory Effect (SME). TEM distinguishes cubic austenite vs monoclinic martensite phases.

SCANNING ELECTRON MICROSCOPY (SEM):

- Uses electron beam (0.3-30 keV) scanned across surface. Resolution ~0.3-3 nm.

- Imaging modes:

  * Secondary electrons (surface topography).

  * Backscattered electrons (atomic number contrast).

  * X-ray analysis (EDX/EDAX for elemental composition).

- Example: SEM images of Mg powders show particle shape and surface composition.

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2. SCANNING PROBE MICROSCOPY (SPM)

- Provides 3D, real-space images with atomic resolution.

- Tools: STM, AFM, NSOM.

SCANNING TUNNELING MICROSCOPY (STM):

- Uses tunneling current between tip and conductive sample surface.

- Resolution: 0.1 nm lateral, 0.01 nm depth.

- Requires conductive/semiconductive samples (can coat insulators with gold).

- Example: IBM STM used to move Fe atoms to form atomic rings.

ATOMIC FORCE MICROSCOPY (AFM):

- Works for both conductors and insulators.

- Tip attached to cantilever; laser measures deflection.

- Modes:

  * Contact mode: Tip touches sample (may damage soft samples).

  * Non-contact: Measures weak forces without contact.

  * Tapping mode: Tip oscillates, reduces damage, useful for biological samples.

- Can measure mechanical properties: adhesion, elasticity, viscoelasticity.

- Example: Surface-treated titanium implants analyzed by AFM for roughness (acid vs alkali treatments).

NEAR-FIELD SCANNING OPTICAL MICROSCOPY (NSOM):

- Combines AFM tip with light, surpasses optical diffraction limit (<50 nm resolution).

- Modes: Transmission, Reflection, Illumination/Collection.

- Useful for studying optical properties of nanomaterials and biological samples.

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3. X-RAY DIFFRACTION (XRD) & SCATTERING

- XRD reveals crystal structure, lattice parameters, crystallite size.

- Bragg's Law: 2d sintheta = lambda

- Scherrer formula for crystallite size: t = 0.9lambda / (B costheta)

- Example: Hydroxyapatite (HA), TCP, TTCP for bone applications. HA bonds directly with bone,

TCP resorbs gradually.

- XRD used to distinguish phases, estimate nanocrystallite size.

SMALL-ANGLE X-RAY SCATTERING (SAXS):

- Scattering at small angles (0-5 degrees), particle size 1-100 nm.

- Reveals shape, size, and porosity of nanostructures.

- Example: HA-filled Polycaprolactone (PCL) composites show decreased crystallinity with filler content.

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4. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR)

- Based on molecular vibrations (stretching and bending).

- Wavenumber (cm-1) used instead of wavelength.

- MIR (400-4000 cm-1) most common for biomaterials.

- Methods:

  * Transmission (KBr pellets for solids).

  * ATR (surface-sensitive, non-destructive).

- Example: FT-IR of HA nanoparticles shows phosphate (PO_4^{3-}) and hydroxyl (OH^-) peaks. Heating increases crystallinity.

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5. DYNAMIC LIGHT SCATTERING (DLS)

- Measures nanoparticle size in suspension using Brownian motion and light scattering fluctuations.

- Principle: Stokes-Einstein equation links diffusion coefficient with hydrodynamic diameter.

- Non-invasive, ensemble technique.

- Useful for nanoparticle sizing (3-3000 nm range).

- Example: Used for in vitro nanoparticle size determination in biological media.

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6. CONTACT ANGLE MEASUREMENTS

- Measures wettability, surface energy, and protein adhesion potential.

- Young's Equation: gammas = gammasl + gammal costheta

- High-energy surfaces (metals) -> low contact angle (hydrophilic).

- Low-energy surfaces (polymers) -> high contact angle (hydrophobic).

- Methods:

  * Static (sessile drop method).

  * Dynamic (advancing/receding angles).

- Example: Water on glass (hydrophilic) vs Teflon (hydrophobic).

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7. MERCURY INTRUSION POROSIMETRY (MIP)

- Measures pore size and porosity using Washburn equation:

  P = -4gamma costheta / D

- Mercury is non-wetting, requires external pressure.

- Detects macropores to mesopores.

- Example: Porous HA ceramics (Endobont) show macropores (~300 um) and micropores (~1.3 um).

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8. GAS ADSORPTION MEASUREMENTS (BET ANALYSIS)

- Measures surface area and porosity using inert gas adsorption (N2, Ar).

- BET isotherm based on multilayer adsorption theory.

- Provides surface area, pore size distribution.

- Example: HA nanorods showed pores ~3.5 nm, BET surface area 42 m2/g.

KEY TAKEAWAYS:

- Optical, electron, and scanning probe microscopes provide complementary imaging scales.

- Spectroscopic (FTIR) and scattering (XRD, SAXS) techniques provide compositional and structural information.

- Surface and porosity characterization (contact angle, MIP, BET) are crucial for biomaterial-tissue interactions.

- No single technique is sufficient - combined methods give complete characterization.