Use of X-ray Diffractometry in Cosmetics

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Introduction

X-ray diffraction, usually abbreviated XRD, is a materials-characterization technique that can identify crystalline phases and provide information about crystal structure, lattice spacing, preferred orientation, crystallite size, and the proportion of some phases in a mixture. It can be valuable in cosmetic research and quality control because powders, pigments, fillers, minerals, and certain active ingredients may exist in crystalline forms. XRD does not, however, prove that an entire cosmetic is safe, identify every ingredient, or measure how much material penetrates skin. Its results must be interpreted with formulation knowledge and complementary analytical methods. (National Institute of Standards and Technology, “X-ray Powder Diffraction”)

Cosmetics as Complex Materials

Cosmetics include lipsticks, powders, creams, lotions, nail products, shampoos, fragrances, and many other preparations intended to cleanse, beautify, or alter appearance. Formulations may contain oils, waxes, polymers, surfactants, water, pigments, preservatives, minerals, and particles. Some are homogeneous liquids; others are emulsions, suspensions, pressed powders, or multiphase solids.

XRD is most informative for crystalline or partly crystalline components. Amorphous polymers, dissolved molecules, oils, and many organic ingredients may produce broad features or weak signals rather than sharp peaks. The method should therefore be selected because the research question concerns phase or structure, not simply because the sample is a cosmetic. (National Institute of Standards and Technology, “X-ray Powder Diffraction”)

Regulatory Context in the United States

The original essay states that cosmetics are regulated by the U.S. Food and Drug Administration and therefore considered safe, then describes the industry as self-regulated. Both statements require correction. Cosmetics are regulated under federal law, and the Modernization of Cosmetics Regulation Act of 2022 significantly expanded FDA authority. Responsible persons must maintain adequate safety substantiation, and facilities and products are subject to new requirements. (U.S. Food and Drug Administration, “Modernization of Cosmetics Regulation Act of 2022”)

Most cosmetic products and ingredients are not individually preapproved by FDA, except for color additives and products that also meet the legal definition of a drug. Manufacturers are legally

responsible for safety and labeling. XRD can support identity and quality evidence but cannot replace toxicology, microbiology, exposure assessment, stability testing, or regulatory review. (U.S. Food and Drug Administration, “Modernization of Cosmetics Regulation Act of 2022”)

Physical Principle of Diffraction

X-rays have wavelengths comparable to distances between atoms in crystals. When an incident beam interacts with an ordered array of atoms, waves scatter in many directions. At particular angles, scattered waves interfere constructively and produce peaks. The pattern of peak positions and intensities is related to the crystal structure and chemical composition of the phase. (National Institute of Standards and Technology, “X-ray Powder Diffraction”)

A crystalline material produces a characteristic pattern that can be compared with reference data. A mixture may show peaks from several phases, although overlap, low concentration, preferred orientation, and matrix effects can complicate identification.

Bragg’s Law

Bragg’s law is commonly written as $n\lambda = 2d \sin\theta$, where λ is the X-ray wavelength, d is the spacing between sets of lattice planes, θ is the incident angle associated with diffraction, and n is an integer order. In a conventional powder diffractometer, results are often plotted as intensity against 2θ. (National Institute of Standards and Technology, “X-ray Powder Diffraction”)

Bragg’s law does not mean that scattering occurs only at a few magical points. All atoms scatter, but the periodic crystal structure causes strong constructive interference under specific geometric conditions. Peak width, shape, and intensity contain additional information, but they are influenced by both the sample and the instrument.

Powder XRD and Single-Crystal XRD

Single-crystal diffraction uses one sufficiently ordered crystal and can determine detailed three-dimensional structures. Powder XRD examines many small crystallites with varied orientations. Cosmetic powders and extracted solid phases are usually more suited to powder diffraction because obtaining a suitable single crystal from a formulation may be impossible.

Powder preparation aims to present many orientations and a flat, representative surface. Grinding may improve randomness but can alter sensitive materials, reduce crystallite size, induce strain, or change polymorphs. Sample handling must therefore match the material.

Instrument Components

A typical laboratory powder diffractometer includes an X-ray source, optics, sample stage, goniometer, detector, shielding, control electronics, and analysis software. Copper K-alpha radiation is common, though other wavelengths may be selected. The instrument scans an angular range while measuring diffracted intensity.

Calibration is essential. Standard reference materials can be used to evaluate peak position, line shape, intensity, and alignment. NIST produces powder-diffraction standards that help laboratories characterize diffractometers. Without calibration, a small peak shift may be incorrectly attributed to the cosmetic sample. (National Institute of Standards and Technology, “Powder Diffraction Standard Reference Materials”)

Sample Preparation for Cosmetic Formulations

The claim that XRD needs no dilution or pretreatment is too broad. A loose face powder may be mounted directly with minimal preparation, but lipstick, cream, lotion, or emulsion may require separation, drying, cooling, or controlled extraction to isolate solids. Each step can change the material. Drying may transform hydrates, melt waxes, or alter polymorphs.

Researchers should document whether they analyzed the intact formulation, a scraped surface, a filtered solid, an ashed residue, or an extracted fraction. Blank containers and substrates may also produce peaks. Replicate sampling is necessary because pigments and fillers may not be uniformly distributed.

Qualitative Phase Identification

Qualitative analysis compares observed peak positions and relative intensities with reference patterns. In cosmetics, possible phases include titanium dioxide polymorphs, zinc oxide, talc, mica, silica-related minerals, calcium carbonate, iron oxides, and crystalline waxes or salts. Identification should use multiple peaks rather than one match.

Elemental composition alone does not determine phase. Titanium and oxygen can occur in rutile or anatase structures with different diffraction patterns. This is a central advantage of XRD over techniques that report only which elements are present.

Quantitative Phase Analysis

With validated models, XRD can estimate the mass fractions of crystalline phases. Rietveld

refinement fits a calculated pattern to the observed data using structural, instrumental, and sample parameters. Internal or external standards may help measure amorphous content. Quantification requires representative sampling and appropriate reference structures.

A result should include uncertainty and detection limits. Minor ingredients may be below detection, and peak overlap can reduce accuracy. Quantitative output from unvalidated software should not be treated as exact merely because it has decimal places.

Crystallite Size, Strain, and Peak Broadening

Small coherent diffraction domains can broaden peaks. The Scherrer equation can estimate crystallite size after correcting for instrumental broadening, but the estimate is not automatically the same as particle size. A particle may contain multiple crystallites, and broadening may also arise from strain, defects, or size distribution.

This distinction matters for nanomaterials. XRD may indicate nanoscale crystallite dimensions for a crystalline ingredient but cannot by itself describe particle shape, aggregation, surface coating, or biological exposure. Electron microscopy, light scattering, surface analysis, and other methods may be needed. (U.S. Food and Drug Administration, “Guidance for Industry: Safety of Nanomaterials in Cosmetic Products”)

Applications to Pigments and Sunscreen Ingredients

XRD can confirm pigment phases and detect changes caused by processing or storage. Titanium dioxide exists primarily as rutile and anatase; formulation performance and surface treatment depend on more than phase alone. Zinc oxide and iron-oxide pigments also produce characteristic patterns. Mineral fillers can influence texture, opacity, slip, and appearance.

Sunscreen analysis requires regulatory precision. In the United States, products intended to prevent sunburn are drugs, even if consumers also view them as cosmetics. XRD can characterize crystalline ingredients but cannot establish sun-protection factor, skin safety, photostability, or exposure.

Counterfeit and Contamination Screening

Counterfeit products may use different fillers, pigments, or crystalline contaminants from authentic products. Pattern comparison can reveal a phase mismatch and support forensic investigation. XRD may also identify unexpected crystalline materials in raw ingredients or finished powders.

It is not a universal counterfeit detector. Two products may share crystalline phases while differing in

dyes, preservatives, fragrance, microbes, metals, or concentration. Chain of custody, validated reference samples, and complementary tests are necessary before drawing legal conclusions.

Nanomaterials and Safety

Nanomaterials may be used to modify transparency, UV behavior, texture, delivery, or stability. FDA guidance emphasizes that safety substantiation applies to nanomaterials as it does to conventionally manufactured ingredients, while recognizing that size, surface, aggregation, and persistence can affect behavior. The presence of a nanoscale phase does not prove harm or safety. (U.S. Food and Drug Administration, “Guidance for Industry: Safety of Nanomaterials in Cosmetic Products”)

XRD cannot measure skin penetration directly. Uptake depends on particle size distribution, coating, vehicle, dose, damaged or intact skin, dissolution, and biological conditions. Claims about penetration require appropriate exposure and toxicological studies.

Complementary Analytical Methods

Scanning electron microscopy can examine morphology; energy-dispersive spectroscopy and X-ray fluorescence provide elemental information; Raman and infrared spectroscopy identify molecular bonds; thermal analysis evaluates melting and phase transitions; chromatography separates organic ingredients; and ICP-MS can measure trace elements. Microbiological testing addresses contamination that diffraction cannot detect.

Using several methods is not evidence that XRD is weak. Each technique answers a different question. Strong characterization begins with a decision tree: phase identity, elemental composition, molecular structure, morphology, concentration, stability, or biological safety.

Quality Assurance and Method Validation

A laboratory method should define the intended use, sampling plan, preparation, scan range, step size, counting time, radiation, calibration, reference database, identification criteria, and acceptance limits. Analysts should use positive controls or known standards and monitor instrument performance over time. (National Institute of Standards and Technology, “Powder Diffraction Standard Reference Materials”)

Validation may examine specificity, precision, repeatability, detection capability, robustness, and accuracy against certified or independently characterized materials. Automated library matches require expert review because background, fluorescence, preferred orientation, and artifacts can produce misleading candidates.

Kinematical and Dynamical Diffraction

Conventional powder analysis usually relies on a kinematical approximation in which scattering events are treated as sufficiently weak and independent. Dynamical diffraction considers multiple scattering and wave-field effects that become important in highly perfect crystals and particular geometries. The original essay correctly notes that the theories can predict different intensities, but most routine cosmetic powders are interpreted using established powder-diffraction methods rather than a general claim that dynamical theory is always superior. (Fewster, 2014)

Choosing the theoretical model depends on crystallinity, thickness, geometry, and analytical objective. Advanced theory should clarify a specific measurement problem rather than appear as an unrelated display of complexity.

Safety in the XRD Laboratory

X-ray instruments use ionizing radiation and require engineered shielding, interlocks, training, controlled procedures, and maintenance. Operators should never defeat safety systems. Chemical hazards may arise during extraction or grinding, and fine powders may create inhalation risk. Laboratory safety applies even when the measurement itself is nondestructive.

“Nondestructive” means the analyzed portion may remain physically available after measurement under certain conditions. It does not mean every preparation leaves the original formulation unchanged.

Conclusion

X-ray diffractometry is valuable in cosmetics when the question concerns crystalline phase, structure, polymorphism, or quantitative phase composition. It can characterize pigments, minerals, fillers, sunscreen ingredients, raw materials, and suspicious phase changes. Accurate use requires representative preparation, calibration, validated identification criteria, and recognition that XRD is insensitive to many amorphous, dissolved, organic, microbial, and toxicological properties. Under modern cosmetic regulation, manufacturers must substantiate safety, but diffraction is only one component of that evidence. Its greatest strength is precise structural information when it is integrated with complementary chemistry, microscopy, exposure assessment, and quality systems. (National Institute of Standards and Technology, “X-ray Powder Diffraction”; U.S. Food and Drug Administration, “Modernization of Cosmetics Regulation Act of 2022”)

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