

Use of X-ray Diffractometry in Cosmetics

Posted on September 30, 2019

Introduction

X-ray diffractometry, commonly called X-ray diffraction or XRD, is a materials-characterization technique that helps identify crystalline substances and examine their structural properties. In cosmetics, the method is useful because many formulations contain crystalline or partly crystalline ingredients such as mineral pigments, titanium dioxide, zinc oxide, talc, mica, and inorganic colorants whose phase composition can influence color, opacity, texture, stability, and performance. XRD does not analyze every cosmetic ingredient equally well. Oils, dissolved molecules, many polymers, and amorphous materials may produce weak or broad diffraction features, so the technique is often combined with spectroscopy, microscopy, thermal analysis, or elemental methods. The physical basis is the interaction of X-rays with ordered atomic planes in a crystal, producing characteristic diffraction peaks at angles related to lattice spacing. NIST defines X-ray powder diffraction as diffraction from planes of atoms in a powdered crystalline sample prepared to represent many possible crystal orientations (National Institute of Standards and Technology [NIST], 2025). In cosmetic analysis, this structural information can support formulation research, quality control, raw-material verification, contamination investigation, and counterfeit screening.

Diffraction Principles and Instrumentation

When monochromatic X-rays strike a crystalline material, atoms arranged in repeating lattice planes scatter the radiation. Constructive interference occurs when the geometry satisfies Bragg's law, commonly written as $n\lambda = 2d \sin\theta$, where λ is the X-ray wavelength, d is the spacing between lattice planes, and θ is the diffraction angle. A powder diffractometer usually contains an X-ray source, optics, a sample holder, a goniometer that controls measurement geometry, and a detector that records intensity as a function of angle. The resulting diffraction pattern acts as a structural fingerprint because crystalline phases produce characteristic peak positions and relative intensities. Powder XRD is particularly suitable for mixtures and routine quality-control samples because finely divided material exposes many crystal orientations to the incident beam. Single-crystal XRD can determine a complete crystal structure when a suitable crystal is available, but that approach is less common for finished cosmetic formulations. Accurate interpretation also depends on calibration, sample preparation, peak overlap, preferred orientation, and instrument resolution rather than on pattern matching alone.

Applications to Cosmetic Ingredients and Formulations

XRD can help distinguish crystalline phases that have the same chemical elements but different structures and therefore different physical behavior. Titanium dioxide is a useful example because anatase and rutile are distinct polymorphs with different diffraction patterns. Zinc oxide, iron oxides, calcium carbonate, talc, mica, and other mineral ingredients can likewise be examined for phase identity, crystallinity, or unexpected components. In pressed powders and mineral makeup, XRD can help verify whether a supplier's material corresponds to the declared crystalline phase. In sunscreen or pigment research, it can complement particle-size, optical, and surface analyses by showing whether processing has changed crystal structure. Quantitative phase analysis is also possible when appropriate standards, refinement methods, and validation are used, although mixtures with amorphous material or overlapping peaks can be challenging. Peak broadening may provide information related to small crystallite size or lattice strain, but instrumental contributions must be separated carefully. XRD therefore works best as one part of a broader analytical strategy rather than as a stand-alone test for overall product safety or quality.

Regulatory Context and Safety Interpretation

Cosmetic regulation in the United States is broader than a simple claim that products are either "FDA approved" or self-regulated. The Modernization of Cosmetics Regulation Act of 2022 substantially expanded FDA's authority and introduced requirements involving facility registration, product listing, serious adverse-event reporting, safety substantiation, and other responsibilities (U.S. Food and Drug Administration [FDA], 2026). XRD can support a manufacturer's quality and safety program by helping characterize raw materials or investigate unexpected crystalline contaminants, but an XRD result does not itself establish that a cosmetic is safe for use. Safety assessment also depends on toxicology, exposure, microbiological quality, chemical purity, use conditions, and applicable restrictions. Nanomaterials require similar caution. XRD may reveal crystal phase and crystallite characteristics, yet it does not provide a complete measurement of particle size distribution, surface chemistry, aggregation, or biological behavior. Electron microscopy, dynamic light scattering, spectroscopy, elemental analysis, and exposure assessment may therefore be needed. Analytical evidence should be interpreted in relation to the actual formulation and the question being investigated.

Sample Preparation, Validation, and

Complementary Methods

Cosmetic samples can be difficult to analyze because finished formulations may contain waxes, oils, polymers, surfactants, pigments, fragrances, and water in complex combinations. Sample preparation must therefore preserve the structural feature being studied while minimizing interference. Powders may be homogenized and mounted carefully to reduce preferred orientation, while creams or emulsions may require separation, drying, or another controlled preparation step if the objective is to examine crystalline solids. Any preparation can alter a formulation, so the analyst should document conditions and avoid assuming that the processed specimen perfectly represents the product in use. Reliable methods also require instrument calibration, reference materials where available, replicate measurements, acceptance criteria, and an understanding of detection limits. XRD can be combined with Fourier-transform infrared spectroscopy, Raman spectroscopy, scanning electron microscopy, thermal analysis, X-ray fluorescence, or chromatographic techniques depending on the question. NIST's long-standing standard powder-pattern work illustrates the importance of well-characterized reference data for phase identification (Swanson et al., 1970). Quality assurance turns a diffraction pattern into defensible analytical evidence.

Counterfeit Screening and Product Development

One practical use of XRD is comparison among raw materials, production lots, or suspected counterfeit products. If a product expected to contain a particular mineral pigment shows an unexpected crystalline phase, the difference may indicate substitution, contamination, manufacturing change, or an analytical artifact that requires further investigation. This approach can be valuable when packaging and appearance alone cannot distinguish authentic material from an imitation. In product development, XRD can also show whether milling, heating, drying, storage, or interaction with other ingredients changes the crystalline form of a component. Such changes may affect texture, color, dissolution, stability, or optical properties. However, absence of a diffraction peak does not automatically prove that an ingredient is absent because low concentration, poor crystallinity, strong matrix interference, or detection limits may conceal it. Likewise, a matching pattern does not prove overall product identity. Analysts should therefore formulate specific questions—such as phase identification or comparative crystallinity—and choose complementary methods for composition, particle morphology, organic ingredients, or trace contaminants.

Conclusion

X-ray diffractometry is a valuable analytical tool for cosmetics when the scientific question involves crystalline structure. It can identify mineral phases, distinguish polymorphs, support raw-material verification, investigate contamination, compare suspected counterfeit products, and monitor structural changes caused by processing or storage. Its usefulness is strongest for crystalline or partly

crystalline ingredients and more limited for oils, solutions, amorphous polymers, and complex organic components. The method relies on characteristic diffraction produced by ordered atomic planes and requires careful sample preparation, calibrated instrumentation, suitable reference data, and appropriate interpretation. In the United States, XRD may contribute to quality and safety substantiation within a broader regulatory framework strengthened by the Modernization of Cosmetics Regulation Act, but it does not substitute for toxicological, microbiological, chemical, or exposure assessment (FDA, 2026). Cosmetic scientists therefore obtain the most reliable conclusions when diffraction data are integrated with complementary analytical techniques. Used in that way, XRD provides structural information that can improve formulation research, manufacturing control, authenticity assessment, and evidence-based investigation of product quality.

References

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