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Seeing the Invisible Lattice: Powder X-ray Diffraction as the Crystallographic Conscience of Dental Materials- A Systematic Review.

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ABSTRACT :

Background: Most materials used in dentistry, such as zirconia, glass-ceramics, calcium-silicate cements, titanium, hydroxyapatite, and the minerals found in enamel and dentin, are crystalline. Their clinical behaviour depends on the types and proportions of crystalline phases present, and how these change during processing, adjustment, and use in the mouth. Powder X-ray diffraction (PXRD) is the main technique for studying these aspects. However, its applications cover many specialties, and reporting practices are inconsistent.

Objective: To systematically review and summarise the use of PXRD in dental research, particularly for prosthodontic materials, and to propose a minimum reporting standard for future studies.

Methods: PubMed/MEDLINE was searched up to October 2026 using eleven specific search strategies that combined "X-ray diffraction" with material and tissue terms. Studies using in vitro, ex vivo, or animal models that reported and interpreted XRD data for dental materials or tissues were included. Information on the material, the purpose of XRD, the quantification method, and the main crystallographic findings was collected and summarised by domain, following PRISMA 2020 guidelines.

Results: The searches returned 1,279 records; 48 primary studies were included. Zirconia ceramics were the most studied domain (n = 14), followed by glass-ceramics (n = 8), calcium-silicate cements (n = 6), dental hard tissues and remineralisation (n = 6), implants and coatings (n = 4), alloys and orthodontic wires (n = 3), bone substitutes, pulp stones and forensic odontology (n = 2 each) and resin composites (n = 1). PXRD was used for five recurring purposes: phase identification, quantification of tetragonal-to-monoclinic phase transformation in zirconia, monitoring crystallisation in glass-ceramics, crystallite size/crystallinity of apatites, and residual-stress analysis. Hydrothermal ageing increased the monoclinic content in 3Y-TZP to about 70%, whereas cubic-rich 4Y/5Y zirconias showed little or no transformation and lacked transformation toughening. Under-firing of lithium (di)silicate left residual lithium metasilicate and markedly reduced strength. Reporting of scan parameters and quantification methods was heterogeneous.

Conclusions: PXRD is essential for connecting the processing, adjustment, and ageing of dental ceramics to their mechanical reliability, and for studying bioactive cements and mineralised tissues. Its surface sensitivity, detection limits for minor phases, and the need for complementary techniques should be considered. A 12-item PXRD reporting checklist is suggested to improve reproducibility.

Keywords: Powder X-ray diffraction (PXRD); Crystallography; Low-temperature degradation; Mineral trioxide aggregate; Dental materials.

Introduction

Clinical dentistry relies heavily on solid-state science. The strength of a monolithic zirconia crown, the translucency of a lithium disilicate veneer, the setting of a calcium-silicate pulp-capping agent, and the acid resistance of enamel all depend on the arrangement of atoms in crystalline lattices. When a technician over-sinters a zirconia framework, a clinician grinds an occlusal contact or sandblasts an intaglio surface, or a restoration remains in a warm, wet mouth for years, the phases within the material can change. Sometimes these changes are invisible, but they can affect fracture resistance.[1]

Since W.H. and W.L. Bragg showed that crystals diffract X-rays in a predictable way,[2] X-ray diffraction (XRD) has become the main tool for identifying crystalline phases and measuring their proportions. In its most common laboratory form, called powder or polycrystalline XRD (PXRD), a monochromatic beam hits a sample with many randomly oriented crystallites. The resulting pattern of peaks serves as a fingerprint for each crystalline phase present. In addition to identification, the positions, intensities, and widths of the peaks provide information on lattice parameters, phase fractions, crystallite size, and residual stress.[3,4]

In dentistry, PXRD is used regularly but is rarely the main focus of a paper. It is usually just one of several techniques used in a materials characterisation study. As a result, the knowledge it provides is scattered across prosthodontics, endodontics, conservative dentistry, implantology, orthodontics, and forensic odontology. Clinicians and postgraduate students often encounter statements such as "XRD revealed 12% monoclinic phase" without knowing how that number was determined, how reliable it is, or what it means in practice.

This systematic review aimed to: (i) summarise the physical basis and quantitative tools of PXRD relevant to dental readers; (ii) identify and map dental studies that used PXRD; (iii) bring together the main findings by material and tissue type, focusing on prosthodontic ceramics; and (iv) point out methodological weaknesses and suggest a minimum reporting checklist.

Fundamentals of PXRD in Dentistry

1 Bragg's law and the diffraction pattern

A crystal is made up of atoms arranged in a repeating three-dimensional pattern, which can be thought of as groups of parallel planes separated by a specific distance. X-rays with wavelength λ scatter off these planes, and the scattered rays reinforce each other only when the additional path length of the deeper ray matches a whole number of wavelengths. This relationship is described by Bragg's law: $n\lambda = 2d \sin\theta$ (Figure 1). Each crystalline phase has its own set of d-spacings, so when the detector scans through different angles (usually shown as 2θ), it creates a unique pattern of peaks for each phase.

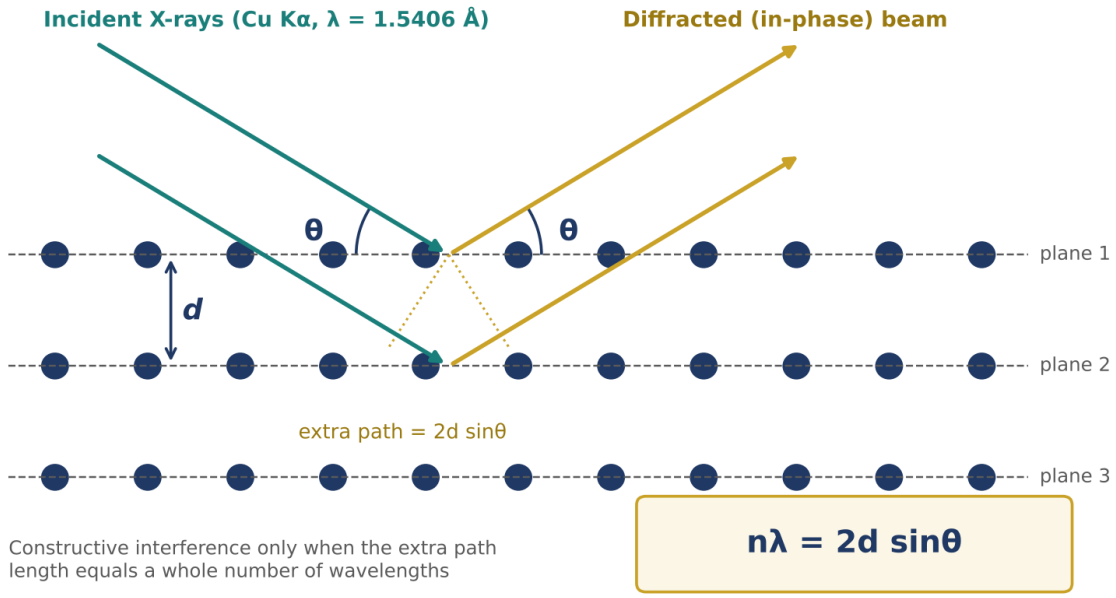


Figure 1. Bragg diffraction happens when X-rays reflect off different layers in a crystal. The reflected waves reinforce each other only if the path difference, 2d sin θ, equals an integer number of wavelengths. Using Cu Kα radiation (λ = 1.5406 Å), each phase produces its own set of 2θ peaks.

Each peak has three features that provide information. The position (2θ) shows which phase is present and, with small shifts, can reveal lattice strain or ionic substitution, such as fluoride or carbonate in apatite. The intensity shows how much of the phase is present and is used for quantitative phase analysis. The width of the peak shows crystallite size and micro-strain. Very small crystallites, like those in biological apatite, produce broad peaks. In contrast, well-crystallised sintered ceramics produce sharp peaks.[3]

2 Instrumentation and specimen preparation

A conventional laboratory diffractometer uses a copper anode (Cu Kα), a goniometer in Bragg–Brentano θ–2θ geometry, and a scintillation or solid-state detector. True powder specimens, which are finely ground to a few micrometres and packed into a zero-background holder, provide the most reliable intensities because the crystallite orientation is random. However, many dental studies analyse flat bulk specimens, such as sintered zirconia discs, glass-ceramic bars, or tooth slabs, to preserve the surface condition produced by grinding, sandblasting, or ageing. This approach is valid and often necessary, but it changes what is being measured. For Cu Kα, the information depth in zirconia is only a few micrometres, so a bulk scan describes just the surface layer rather than the entire restoration.1 The studies included also used micro-XRD for small or curved regions, synchrotron XRD for high resolution and strain measurement, and grazing-incidence XRD for thin coatings (Figure 2).

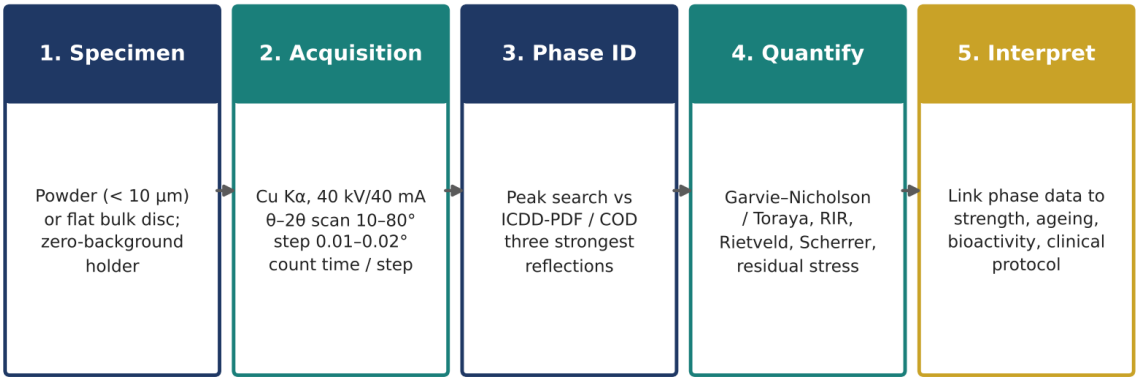


Figure 2. Typical PXRD workflow in a dental-materials study, from specimen preparation to clinical interpretation.

3 Quantitative tools

Table 1 shows the quantitative relationships most often used in the studies discussed. For zirconia, the monoclinic fraction is typically calculated from the integrated intensities of the monoclinic (−111) and (111) reflections and the tetragonal (101) reflection using the Garvie–Nicholson relation,[5] and then converted to a volume fraction using the Toraya calibration.6 Rietveld refinement matches the whole calculated pattern to the measured one and is now considered the most rigorous method for multiphase materials, such as cubic-containing zirconias and lithium (di)silicate glass-ceramics.[4]

Parameter	Expression / method	Dental application
Phase identification	Match d-spacings / 2θ of the three strongest peaks to ICDD-PDF or COD reference cards	Confirms constituents of MTA, sealers, glass-ceramics, coatings, pulp stones
Monoclinic fraction (Xm)	Xm = [Im(−111) + Im(111)] / [Im(−111) + Im(111) + It(101)] [5]	Zirconia ageing (LTD), grinding, airborne-particle abrasion
Monoclinic volume (Vm)	Vm = 1.311Xm / (1 + 0.311Xm)6	Corrects Xm for non-linear intensity response
Crystallite size (D)	Scherrer: D = Kλ / (β cosθ), K ≈ 0.9, β = peak breadth in radians3	Apatite in enamel/dentin, remineralisation, burnt teeth, HA coatings
Crystallinity index	Ratio of crystalline peak area to total (crystalline + amorphous) area	Bone substitutes, demineralised/remineralised dentin, MTA portlandite
Full-pattern quantification	Rietveld refinement of structure and scale factors [6]	Cubic/tetragonal/rhombohedral zirconia; Li2Si2O5 vs Li2SiO3; residual glass
Residual stress / lattice strain	sin2ψ method or peak shift relative to stress-free reference	Implant surfaces, ATZ composites, cement-induced dentin strain

Materials and Methods

1 Protocol and reporting

This review followed the PRISMA 2020 statement.[7] Since the evidence comes from laboratory studies with different designs and outcomes, we planned a qualitative (narrative) synthesis instead of a meta-analysis from the start.

2 Focused question (PICOS-adapted)

Population/specimens: dental restorative, prosthodontic, endodontic, implant, and orthodontic materials, as well as human dental hard tissues and calcifications.

Intervention/exposure: characterization using powder or polycrystalline X-ray diffraction, including micro, synchrotron, and grazing-incidence methods.

Comparison: untreated, as-received, or control specimens when relevant.

Outcomes: phase composition, phase transformation, crystallite size, crystallinity, lattice strain or residual stress, and their relationships to mechanical, biological, or clinical outcomes.

Study designs: in vitro, ex vivo, and animal studies, as well as clinical studies that include an XRD component.

Focused question: For dental materials and tissues, what information has PXRD provided, and how has it helped explain processing, aging, and clinical behavior?

3 Information sources and search strategy

We searched PubMed/MEDLINE up to October 2026. Instead of using a single broad query, we applied 11 domain-specific strategies to identify studies in which XRD was a secondary technique. Table 2 lists these strategies and their results.

Table 2.Search strategies and yields (PubMed/MEDLINE, October 2026)

Search string (PubMed)	Records
"X-ray diffraction" AND zirconia AND (monoclinic OR "phase transformation") AND dental	295
"X-ray diffraction" AND ("lithium disilicate" OR "glass-ceramic") AND crystalline phase AND dental	41
"X-ray diffraction" AND ("mineral trioxide aggregate" OR "calcium silicate cement" OR Biodentine)	217
"X-ray diffraction" AND (enamel OR dentin) AND (crystallinity OR hydroxyapatite crystallite)	255
"X-ray diffraction" AND dental implant AND (hydroxyapatite coating OR titanium surface OR anatase)	372
"X-ray diffraction" AND ("dental calculus" OR "salivary calculi" OR "pulp stones")	56
"X-ray diffraction" AND (bone substitute OR "bone graft") AND (deproteinized bovine OR xenograft) AND crystallinity	10
"X-ray diffraction" AND (nickel-titanium OR NiTi) AND orthodontic wire AND phase	10
"X-ray diffraction" AND (burned OR cremated OR incinerated) AND teeth AND forensic	6
"X-ray diffraction" AND cobalt-chromium AND (selective laser melting OR casting) AND dental	13
"X-ray diffraction" AND (CAD-CAM OR "3D-printed") AND (denture base OR resin composite) AND filler	4
Total records identified (before de-duplication)	1279

4 Eligibility criteria

Studies were included if they were original research on dental materials or human dental tissue that presented and interpreted XRD data and were published in English. Exclusion criteria comprised reviews, editorials, and conference abstracts; studies in which the substrate was not a dental material or tissue (for example, construction cements or orthopaedic bone cements); and studies that mentioned XRD without reporting its results.

5 Study selection, data extraction and synthesis

Records were screened by title and abstract, followed by assessment of the full report against the eligibility criteria. For each included study, the following data were extracted: material or tissue, XRD variant and purpose, quantification method (Garvie–Nicholson, Toraya, Rietveld, Scherrer, or other), principal crystallographic finding, and any correlation with mechanical, biological, or clinical outcomes. Studies were grouped into ten domains and synthesised narratively.

6 Methodological appraisal

No validated risk-of-bias tool exists for crystallographic characterisation. Therefore, each study was appraised descriptively for the transparency of XRD reporting, including the instrument, radiation source, scan range, step size, specimen form, reference database, and quantification method. These observations informed the reporting checklist proposed in Section. [5]

Results

1 Study selection and characteristics

The eleven searches identified 1,279 records. After screening and eligibility assessment, 48 primary studies met the inclusion criteria (Figure 3); studies on non-dental cement and bone cement substrates were excluded. Eight methodological and standards sources were additionally used to define the analytical framework. The distribution of included studies across domains is shown in Figure 4, and their characteristics are summarised in Table 3.

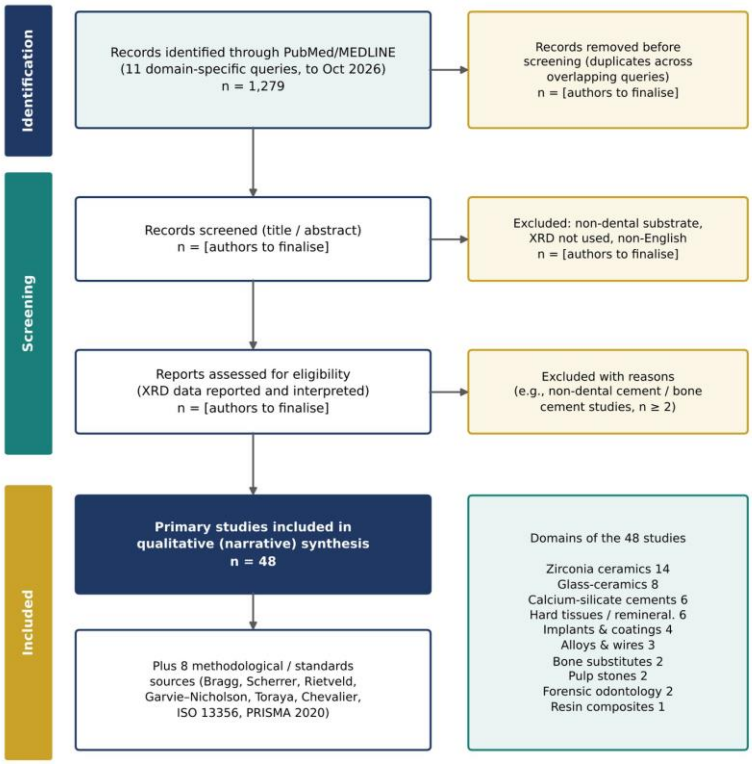


Figure 3. PRISMA 2020 flow diagram. Identification count is from the executed searches; intermediate screening counts are to be finalised after duplicate independent screening by the review team.

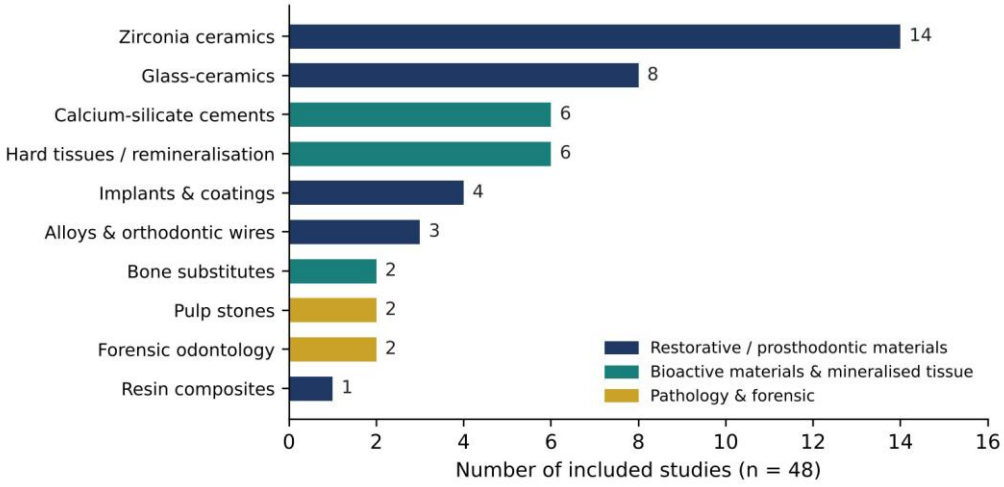


Figure 4. Evidence map: distribution of the 48 included studies across application domains.

The majority of studies (n = 30) investigated restorative and prosthodontic materials. In only a limited number of studies was XRD the primary outcome; more commonly, it was employed in conjunction with three to six complementary techniques, including scanning electron microscopy (SEM), energy-dispersive spectroscopy, Raman or FTIR spectroscopy, and mechanical testing.

2 Zirconia ceramics: the tetragonal–monoclinic story

Yttria-stabilised zirconia derives its toughness from the stress-induced transformation of metastable tetragonal (t) grains into the monoclinic (m) phase, accompanied by an approximately 4% volume increase that impedes crack propagation.

When this transformation is initiated by water at intra-oral temperature, it is referred to as low-temperature degradation (LTD) or hydrothermal ageing. This process progresses from the surface inward, increases surface roughness, and can ultimately reduce material strength.1 PXRD is the standard technique for detecting and quantifying this transformation (Figure 5), and ISO 13356 for zirconia implant ceramics mandates an XRD-based accelerated-ageing assessment.[8]

A. Ageing of conventional 3Y-TZP

Autoclave ageing (134 °C, 2 bar, 24 h) increased the monoclinic content of two commercial 3Y-TZPs from 0% to 66–71%. Despite this, biaxial flexural strength increased, indicating that early transformation induces protective compressive stresses prior to the onset of roughening and microcracking.[9] Pandoleon et al. used XRD, XPS, and FTIR to demonstrate that the t→m transformation was associated with yttrium depletion and oxygen loss.

These findings support a mechanistic model in which water-driven destabilisation creates vacancies at the surface.[10] Additionally, exposure to ionising radiation at levels used in head-and-neck radiotherapy (70 Gy) resulted in a 7% increase in the monoclinic content of translucent zirconia.[11]

B. Translucent 4Y/5Y zirconias and the cubic phase

Higher-yttria grades contain a substantial cubic phase, which enhances translucency but does not undergo transformation. PXRD consistently confirmed their superior ageing resistance: zirconias with more than approximately 30 vol% cubic phase exhibited no monoclinic peaks after autoclaving.12 Furthermore, 5Y zirconia demonstrated significantly greater LTD resistance compared to 3Y.[13] However, this improvement comes at the cost of reduced strength and toughness. Harada et al. reported characteristic strengths of approximately 620 MPa for 5Y and 950 MPa for 3Y zirconia, and observed that even a small monoclinic fraction in fine-grained regions of 5Y decreased reliability.[13] In four-unit monolithic fixed dental prostheses, ageing and chewing simulation increased monoclinic content in 3Y and 4Y frameworks but not in 5Y; the lack of transformability in 5Y was associated with cracks at abutment walls and margins, prompting the authors to advise against the use of 5Y-PSZ for four-unit FDPs.[14] Ageing responses varied among 3Y, 4Y, and 5Y compositions in both surface and bulk properties.[15]

Rietveld refinement has provided a more detailed understanding of these materials. In two 5Y zirconias, the predominant phase was pseudocubic (66-81 mass%). Airborne-particle abrasion generated approximately 37 mass% of the rhombohedral phase, whereas LTD resulted in only 0.9–2.8 mass% of the monoclinic phase.[16] These distinctions are not detectable using the simple Garvie–Nicholson ratio, which assumes a two-phase t/m system.

C. Laboratory and chairside procedures

PXRD has documented how routine procedures alter zirconia surfaces. Hydrofluoric acid alone produced minimal transformation (0.8%), but liner application with HF produced 8.7%; unveneered zirconia aged to 70.25% monoclinic, whereas veneering porcelain applied by layering (4.41%) or pressing (11.57%) protected the framework.[17] Finishing and polishing systems differed in the degree of partial transformation they induced, with the smoothest protocols also producing the least biofilm.[18] In implant applications, XRD confirmed coexisting monoclinic and tetragonal phases on UV-treated zirconia,[19] and accelerated ageing of Y-TZP dental implants produced limited degradation that did not reproduce the failure pattern of retrieved orthopaedic femoral heads.[20]

D. Alumina-toughened zirconia (ATZ)

XRD-based residual-stress analysis revealed that ATZ exhibited higher compressive residual stresses compared to 3Y-TZP. Although both materials underwent transformation after ageing, ATZ experienced less strength loss.[21] Subsequent studies on experimental ATZ demonstrated the presence of the monoclinic phase after both autoclave and hydrothermal-reactor ageing, yet ATZ maintained greater microstructural stability than 3Y-TZP, supporting its suitability for long-span frameworks.[22]

3 Glass-ceramics: watching crystallisation happen

For glass-ceramics, PXRD addresses whether the intended crystalline phase has formed in the desired quantity. CAD-CAM lithium disilicate is initially milled in a partially crystallised lithium metasilicate (Li2SiO3) state and subsequently converted to lithium disilicate (Li2Si2O5) during crystallisation firing. Using a combination of DSC, XRD, and SEM, Lien et al. identified three distinct microstructural stages and demonstrated that rapid lithium disilicate growth commenced only after metasilicate formation was complete.[23] Firing at 820 °C, rather than the recommended 840 °C, resulted in residual metasilicate and glass, reducing flexural strength by approximately 71%, highlighting the clinical significance of precise furnace calibration.[24]

Quantitative XRD has demonstrated that not all "lithium silicates" possess identical properties. Suprinity PC retained a substantial fraction of lithium metasilicate after crystallisation, whereas IPS e.max CAD developed surface cristobalite. In both materials, cracks introduced by CAM grinding were healed during crystallisation firing.[25] In Obsidian, the metasilicate fraction decreased from 41 to 37 vol% with the formation of approximately 6 vol% lithium phosphate, accompanied by an increase in fracture toughness from 1.63 to 1.84 MPa√m.[26] Following clinical adjustment, XRD was employed to characterise the crystalline phases of heat-pressed lithium disilicate subjected to etching, refiring, diamond adjustment, and air abrasion.[27]

Other systems include leucite-reinforced veneering porcelain, where leucite is the only crystalline phase. Adding silver vanadate introduces a second microcline phase.[28] There are also machinable calcium-mica/fluorapatite glass-ceramics,[29] as well as wollastonite and cristobalite glass-ceramics, which

have been suggested for one-piece implants.[30]

4 Calcium-silicate cements and root-canal sealers

One of the earliest dental uses of PXRD in this area showed that ProRoot MTA and Portland cement share the same main phases: tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite. In MTA, bismuth oxide is added as a radiopacifier.[31]PXRD later became the standard tool for checking the composition and setting products of new calcium-silicate materials. Four commercial cements exhibited similar patterns, even though their particle sizes and cytocompatibilities differed.[32] The hydration product portlandite (calcium hydroxide), which is responsible for the alkalinity and bioactivity of these cements, was most abundant and most crystalline when MTA set at neutral pH. It was least abundant in acidic conditions, which is relevant to inflamed periapical tissues.[33]

For sealers, XRD showed calcium hydroxide peaks after setting in an experimental tricalcium silicate sealer and in Apexit Plus, but not in MTA Fillapex.[34] Premixed bioceramic sealers contained tricalcium silicate, dicalcium silicate, and zirconium oxide, but did not produce detectable calcium hydroxide, even though calcium was released into solution.[35] XRD also helped explain why resin-modified TheraCal LC reacts more slowly than water-based Biodentine and Retro MTA.[36]

5 Enamel, dentin and remineralisation

Biological apatite is a non-stoichiometric, carbonate-substituted hydroxyapatite with nanometre-sized crystallites, resulting in broad diffraction peaks. XRD, along with density-functional modelling, showed that chemical gradients within enamel crystallites, such as a magnesium-rich layer surrounding a core rich in sodium, fluoride, and carbonate, create residual stresses. These stresses may explain why the core dissolves more easily in acid.[37]

In remineralisation research, XRD and Raman spectroscopy showed that topical application of 5% NaF reduced dentin crystallite size and crystallinity. In contrast, cathodal iontophoresis increased the mineral's crystallinity and formed fluorohydroxyapatite.[38] Using the Scherrer equation, researchers found that hydroxyapatite-based endodontic sealers led to immature, poorly crystalline apatite in cervical radicular dentin.[39] Zinc-doped polymeric nanoparticles increased crystallinity and strengthened apical dentin.[40] Radiotherapy at 60 Gy caused only a slight decrease in the XRD peaks of cervical inner enamel.[41]

In prosthodontics, synchrotron XRD showed that dentin had greater lattice strain with dual-cure resin cement than with resin-modified glass ionomer cement. Postoperative sensitivity was also higher with the resin cement immediately after cementation, but there was no significant link between lattice strain and sensitivity.[42]

6 Implant surfaces, coatings and bone substitutes

XRD confirmed that calcium-phosphate coatings on titanium have a composition similar to bone mineral.[43] It also showed that an α-tricalcium phosphate film made with a pulsed Er:YAG laser turned into hydroxyapatite in artificial saliva.[44] XRD was also used to study chitosan and hydroxyapatite coatings on stainless steel.[45] For hybrid titanium implants, XRD residual-stress analysis found compressive stresses of −201.2 MPa on the rough part and −20.2 MPa on the smooth part. This difference created a galvanic potential at the interface, where pitting was observed.[46]

Substitutes: Increasing the deproteinisation temperature of bovine bone increased crystallinity, as observed by XRD, implying slower in vivo resorption. Therefore, processing temperature can be used to tailor graft behaviour.[47] A porous phosphate-glass graft was confirmed to be amorphous.[48]

7 Dental alloys and orthodontic wires

In metals, XRD distinguishes allotropic and martensitic phases. Micro-XRD detected martensite in Sentalloy NiTi wires after combined thermocycling and bending, consistent with reduced stability of the austenite–martensite transformation under simulated intra-oral conditions.[49]

Stainless-steel archwires contained both austenite and martensite, while NiTi wires were mostly austenitic.[50] In selectively laser-melted cobalt–chromium, the annealing temperature altered the ε-phase and second-phase precipitates observed by XRD. These changes affected how well the metal bonded with ceramic, and samples annealed at 1150 °C had the strongest bonds.[51]

8 Pathological calcifications and forensic odontology

XRD showed that pulp stones are composed of partially carbonated apatite,[52] which has a structure similar to that of nearby dentin but contains more copper and zinc.53 In forensic odontology, XRD can be used on whole teeth without causing damage. When teeth are burned, the size of hydroxyapatite crystals grows as the temperature rises, which may help estimate how hot the fire was.[54] Peer and Sarig found that heating teeth to about 400 °C made dentin apatite crystals much smaller, while heating to about 700 °C slightly increased enamel crystal size and reduced dentin volume by up to 36%.[55]

9 Resin composites

Resin-based materials are mostly amorphous, so XRD is not often used. However, it was used to study the surfaces of CAD-CAM and direct resin

composites, and research found that less biofilm formed on CAD-CAM blocks.56

Discussion

1 Principal findings

This review identified five recurring roles for PXRD in dentistry: phase identification; phase-transformation quantification in zirconia; crystallisation monitoring in glass-ceramics; crystallite size and crystallinity of apatitic tissues and grafts; and residual stress or lattice strain measurement. Across these roles, the technique's value lay less in the numbers themselves than in the links it established among processing, chairside handling, ageing, and clinical performance.

2 Relevance to prosthodontic practice

For prosthodontists, the findings about zirconia and glass-ceramics have clear implications. First, using highly translucent 4Y/5Y zirconias means giving up some transformation toughening in exchange for better ageing resistance. PXRD shows these materials hardly transform, which explains why they are weaker and less resistant to damage.[12–14] When choosing materials for long-span or high-stress posterior FDPs, this trade-off should be considered. Second, procedures such as airborne-particle abrasion, grinding, and polishing alter the crystalline surface layer.[16–18] If adjustments are needed, it is best to polish systematically and, if the manufacturer recommends it, use regeneration firing. Third, lithium (di)silicate restorations require a complete and properly calibrated crystallisation firing. If the temperature is 20 °C too low, residual metasilicate remains, and strength drops sharply.[24,25] Regular furnace calibration is therefore essential for quality assurance, not just a technical detail.

3 Methodological heterogeneity

Reporting varied a lot. Many studies did not specify the scan range, step size, count time, the reference database for phase matching, whether specimens were powders or bulk surfaces, or whether monoclinic content was measured as a Garvie–Nicholson intensity ratio, a Toraya-corrected volume fraction, or a Rietveld mass fraction. These methods are not interchangeable. As a result, values such as "10% monoclinic" from different labs are difficult to compare, and using a two-phase t/m model without accounting for cubic or rhombohedral zirconias can yield a misleading picture of the actual phase composition.[5,6,16]

4 Limitations of PXRD and complementary techniques

PXRD has some built-in limitations that dental researchers should keep in mind. In dense ceramics, it provides information only from a few micrometres deep, so it cannot show how far the transformation has progressed unless you use cross-sectioning or other methods. Minor phases below about 1–5 wt% might not be detected, and amorphous content, such as residual glass in glass-ceramics or amorphous calcium phosphate in early remineralisation, appears only as a broad hump unless an internal standard is used. Factors like preferred orientation, peak overlap, and curved or rough clinical specimens can also distort the results.

5 Future directions

Several advances are expected to broaden the use of PXRD in dentistry. These include in situ and temperature-resolved XRD for tracking crystallisation and sintering as they happen, grazing-incidence XRD for non-destructive depth profiling, and routine Rietveld analysis for multiphase translucent and gradient zirconias. XRD is also being used to study additively manufactured zirconia, glass-ceramics, and Co-Cr, where post-processing affects phase content.[51] Machine learning is starting to help with phase identification. To combine data from different studies, standardised reporting will be crucial.

6. Limitations of this Review

This review only searched PubMed/MEDLINE. Including Scopus, Web of Science, and Embase would likely have yielded more studies, especially from materials science journals. Since XRD is often a secondary method, it may not be mentioned in titles, abstracts, or MeSH terms, so some relevant studies may have been missed. Most of the included studies were in vitro and used different protocols, making meta-analysis impossible. There is no validated tool to assess risk of bias in crystallographic characterisation. Also, the de-duplication and screening numbers in the PRISMA diagram should be checked by an independent reviewer before submission.

Conclusions

Given the limitations of this systematic review, these conclusions can be made:

1.PXRD is the main method for identifying and measuring crystalline phases in dental materials and tissues. It has been used in ten different areas of dentistry.

2. For zirconia, PXRD has shown that hydrothermal ageing, irradiation, adjustment, and surface treatment change the balance between tetragonal and monoclinic phases. Cubic-rich translucent zirconias forgo transformation toughening in exchange for improved ageing resistance.

3. In lithium (di)silicate glass-ceramics, PXRD has shown that complete crystallisation is important for mechanical performance, and that commercial products vary in their remaining phases.

4. For calcium-silicate cements, mineralised tissues, coatings, and grafts, PXRD helps determine composition, hydration products, crystallinity, and crystallite size. It also supports forensic estimates of heat exposure.

5. Inconsistent reporting makes it hard to compare studies. Using a minimum reporting checklist, such as the proposed PXRD-Dent, is recommended.

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