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**Comparative Mineralogical Analysis of Brazilian Portland Cements Using Two Different Approaches; Conventional and High-resolution Synchrotron X-ray Diffractometries.**

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Mineralogical analysis of Portland clinker and cements using X-ray diffraction methods, coupled with Rietveld analysis, is crucial for assessing the quality and performance of these materials directly at the production facility. This analytical approach allows for the precise identification and quantification of all crystalline phases present, providing vital insights into the material's compositional integrity. By understanding the mineralogical composition, manufacturers can optimize the clinker and cement production process, ensuring consistent product quality. Moreover, this method aids in the early detection of potential issues, facilitating timely corrective actions to maintain high standards of product performance and reliability. Eight distinct samples provided by the Brazilian Association of Portland Cement (ABCP)—Clinker, CPI-40, CPIIE-32, CPIIF-32, CPIIZ-32, CPIII-40RS, CPIV-32, and CPV-ARI—were subjected to comparative analysis. Two diffractometry methods were employed: a conventional laboratory Thermo Fisher ARL X'TRA Companion X-ray Diffractometer and high-resolution synchrotron X-ray diffractometry at the European Synchrotron Radiation Facility (ESRF) beamline ID31. For the conventional diffractometry, Bragg-Brentano geometry in reflection mode with spinning and Cupper radiation was used. For the synchrotron measurements, the sample powders were loaded into cylindrical slots (approx. 1 mm thickness) held between Kapton windows in a high-throughput sample holder. Each sample was measured in a transmission with an incident X-ray energy of 75.00 keV (? = 1.653 nm). Measured intensities were collected using a Pilatus CdTe 2M detector (1679 × 1475 pixels, 172 × 172 µm2 each) positioned with the incident beam in the corner of the detector. The sample-to-detector distance was approximately 1.5 m for the high-resolution measurements and 0.3 m for the total scattering measurement. Background measurements for the empty windows were measured and subtracted. NIST SRM 660b (LaB6) was used for geometry calibration performed with the software pyFAI followed by image integration including a flat-field, geometry, solid-angle, and polarization corrections. Each sample underwent a detailed Rietveld refinement using TOPAS v. 6 to determine the mineralogical composition and integrity, vital for assessing the quality and performance of cement products. The results indicated a high correlation and statistical agreement between the laboratory diffractometry and the synchrotron method. The findings demonstrate the importance of in-factory mineralogical analysis using X-ray diffraction and Rietveld analysis. The cross-validation between laboratory and high-resolution synchrotron methods ensures the accuracy of mineralogical assessments, which is imperative for optimizing production processes, ensuring product consistency, and maintaining high standards of product performance and reliability in the cement industry.