METHODS OF SAMPLE PREPARATION IN X-RAY DIFFRACTOMETER (XRD)

Anchit Jain[1], Dr. N. K. Dhapekar[2]

[1]Student, Department of civil Engineering, Kalinga University, Naya Raipur, Chhattisgarh, India.

[2]I/C Head, Department of civil Engg. Kalinga University, Naya Raipur, Chhattisgarh, India.

Abstract

Proper sample preparation is crucial in getting highly quality X-ray diffractometer data. If the sample has not been putted within the parameters to properly prepare XRD sample then it introduces errors that make phase identification difficult to impossible and estimates of abundances and crystallinity erroneous. Ideally, we might prefer to lead three conditions so as to own good data: (a) Total randomness of crystallite orientation. (b) Sufficient number of crystallites to induce a representative intensity distribution for the sample. (c) Sufficient diffraction intensity to satisfy counting statistics. This research paper highlights the methods of perfect XRD sample preparation of solids, liquids and gases. The structurally changed materials in response to solid-gas interaction possesses great importance in many areas. The X-ray structures of liquid sodium, potassium and sodium-potassium alloys are investigated by an improved method. By employing a reservoir technique a plane surface of liquid metal has been examined by a monochromatic X-ray beam in an exceedingly focusing diffractometer.

Keywords: X-ray diffractometer, crystallinity, sample, methods.

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1. Introduction:

The preparation of samples is one among the critical steps in many analytical methods, and particularly for X-ray powder diffraction (XRD) analysis. Reckoning on the character of the powder XRD study and therefore the specified results, sample preparation will be of minimal importance or it will be crucial. If an analysis of a simple, familiar material is desired, the fabric are often quickly ground in an exceedingly hand mortar and pestle and packed into a cavity-type sample holder, paying no attention to orientation or particle-size effects. On the opposite hand analyses of complex materials involving search/match procedures, quantitative phase analysis, lattice-parameter refinement, or Rietveld refinement of crystal structures require rather more careful attention to the strategy and execution of sample preparation and mounting. The 2 most important results desired in these more complex cases are accurate and precise line positions and intensities. An analysis as simple as a manual or computer search/match on an unknown will be way more successful if accurate data are obtained. Minimizing the systematic sample-related effects is as important as accounting for instrumental systematic errors within the collection of accurate and precise data.

2. Literature Review:

Prof. N.K. Dhapekar[1], Prof. A.S. Majumdar[2] and Dr. P. K. Gupta[2], "Study of Phase Composition of Ordinary Portland Cement Concrete Using X-Ray Diffraction" [1] during this research paper, an experimental study is performed on powder ordinary cement concrete samples using X- ray diffraction (XRD) which jambs a assured approach for phase composition of concrete. Laboratory X-ray diffractometer was used for XRD analysis of concrete samples. The potential presence of cement content in hardened ordinary hydraulic cement concrete has been determined by diffraction analysis. They need found that this approach may replace the traditional analysis of hardened concrete which is tedious and time consuming. During this paper an effort has been made to quantify the phases present in ordinary Portland cement concrete. The results of phase quantification obtained from XRD analysis has shown good agreement with the experimental values and it's been concluded that XRD techniques may sway be an efficient tool for phase composition in practice to widen the knowledge concerning the concrete used.

Paul Monceyron Røren[1], Kristoffer W. B. Hunvik[2], Vegard Josvanger[3], Ole Tore Buseth[4] and Jon Otto Fossum[4] "Controlled Sample Environment for studying solid– gas interactions by in situ powder X-ray diffraction" [2] during this research paper, a cell of sample for X-ray diffraction studies in powder form with in place applied pressure and control of temperature is incontestable. The cell relies on a previously reported design and consists of a glass or quartz capillary glued into a Swagelok weld gland; this configuration can inhibition to 100 bar (1 bar = 100 KPa). The cell is placed connected with a copper plate for control of temperature between -30 and 200C. This can be often achieved by Peltier elements, heat cartridges and a refrigerated circulating bath. Commissioning tests were performed during a custom-made small/wide-angle X-ray diffractometer at the Norwegian University of Science and Technology. The system is well portable to synchrotron facilities.

Prof. N. K. Dhapekar[1] and Prof. D. M. Chopkar[1] "Structural Health Monitoring of Ordinary Portland Cement Concrete Structures Using X-Ray Diffraction" [3] In Ordinary Portland cement concrete structural health monitoring has significant interest in life questions of safety. This approach of structural health monitoring by XRD has been observed as an alternate to numerous instrumentation methods. During this paper using advance software package it absolutely was possible to urge reliable results of phase composition and compressive strength of cement concrete structure. Because the standard assurance of concrete structures is more and more becoming an important concern, the authors are concluded that diffraction technique incorporates a large prospective to grapple up with such concerns without detriment of the structural members and influencing the structures in satisfactory condition for the client. This method can also be applied to concrete mixes which contains aggregate or admixtures and liberate soluble silica under the condition of the analysis like slag, sodium, silicate etc.

3. Methodology:

a) Hand Grinding:

Usually powdered XRD samples are prepared by hand grinding employing a mortar and pestle. The mortar and pestle is created out of a variety of materials like agate, corundum, or mullite. The kind of employed material will depend upon the hardness of the sample compared to the material of the mortar and pestle and whether contamination introduced by the grinding medium will effect subsequent interpretations of knowledge. Typically samples are ground under a liquid medium like ethanol or methanol to attenuate sample loss during grinding and to mitigate structural damage to the phases in sample which is able to be caused by grinding. It can however be tedious and physically demanding handy grind samples, especially if sample is extremely hard or has other physical properties making it difficult to grind.

b) Mechanical Grinding:

Though it's possible at hand grind a sample to 1 μm, as mentioned above, it's tedious and difficult. Mechanical grinders are an exquisite because of grind the sample to grain sizes approaching 1 μm for quantitative XRD work. This can be through with shatter boxes or ball mills, but both of these produce an oversized particle size distributions. McCrone Mills are the most effective to provide both small grain sizes and narrow size distributions. They include a Teflon cup holding pellets manufactured from either agate corundum, or tungsten carbide which is then loaded into a holder that shakes the pellets at a high frequency for a set amount of your time. Again, a liquid medium like ethanol is used to chop back lattice stain during milling. The draw backs of this method is simply a little low amount of sample are processed and there's always slightly bit of contamination (< 1 wt. %).

4. Conclusion and Practical Applications:

- Laboratory XRD can be effectively used for qualitative phase composition analysis of a sample which determines the relative amounts of phases in a mixture by referencing the relative peak intensities.
- Finely grinded powder sample indicates crystalline size and macrostrain by peak broadening and also helpful in measuring other defects by analysing peak shapes and peak width.
- It can be widely used to determine unit cell lattice parameters, index positions and also bravais lattice symmetry. Lattice parameters can alter and thus gives statics about alloying, doping, strains etc.
- XRD is useful in determining residual stress and residual Strain or macrostrains.
- Better sample provides satisfactory results of crystal structure by rietveld refinement of the entire diffraction pattern.

- Complicated inorganic and organometallic systems have been analysed using single crystal methods through XRD.

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