SOME OF THE PHYSICAL, PHYSICOCHEMICAL AND TECHNOLOGICAL PROPERTIES OF POWDER MATERIALS AND THEIR RELATION TO THE X-RAY DIFFRACTION

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Abstract

Relations of some properties of powders to the X-Ray diffraction are mentioned. The properties are specific density, bulk volume, moisture, specific surface, character of the surface, rheological properties, sieve residue, morphology of particles, particle size distribution, dispersibility, content of water soluble salts and rheological properties of powder suspensions.

Keywords
X-ray diffraction, powders, powder materials

Density can be determined by the X-ray diffraction from the lattice parameters and mass of the elementary cell. It has some advantages from the density bottle determination. Specific density of individual phase can be determined by the X-ray diffraction even from the phase mixture and there is no need to separate single phases.

Bulk volume affects sample preparation. Samples with high bulk volume have lower sample consumption.
Specific surface. Powders with high specific surface usually have high bulk volume and affect sample preparation in similar way. Such powders consists of very small particles, often with disordered structure. It results in line profile broadening, worse separation of the background and in such way in lower accuracy of the intensity determination and higher detection limit of phase analysis. Typical example of such materials are catalyst supporters because high specific surface is necessary for their efficiency.

Moisture content ought to be regarded with quantitative phase analysis. Not considerate it means to lower precision of analysis. There is also trouble with some moisture samples preparation. Moisture during the measurement is escaping and the sample can be spilled.

Surface character impact on originating of static electricity which can make some troubles with sample preparation.

Surface treatment affects character of the surface. It serves for hydrophobization or hydrophilization of the surface. Its content is usually low, layer on the particle surface is thin and in such way is not detectable by X-ray diffraction. High surface treatment can affect results of quantitative phase analysis.

Sieve residue can be determined in watered way on the sieves of 0.063 or 0.040 mm mesh. X-ray diffraction can distinguish if the residue consists of primary particles or agregates of them.

Morphology of particles is related to the texture and in such way impacts on results of quantitative and in some extend even on qualitative phase analysis.

Particle size distribution is determined with the help of sedimentation methods (Andreasen pipette or ultracentrifuge) or optical methods (optical or electron microscopy). X-ray diffraction can catch the large particles from the film detection and the small ones from the diffraction line broadening. In contrast to optical or sedimentation methods, diffraction is suitable for size determination of primary particles, which are mostly identical with coherent areas.

Dispersibility is defined as degree of grinding in certain binding agent. It is determined with grindometer. X-Ray diffraction can sometimes help with explanation of bad grinding. If large coherent particles are found, the reason of the bad grinding can be promoted by the content of coarse particles in pigment or filler.

Content of water soluble salts. This content is mostly too low for X-Ray diffraction. However, evaporation residue can be used for qualitative phase analysis.

Colour is a psychosensoric quality. It is determined by spectral reflectivity of the surface and by the spectral sensitivity of human eye in the range of visible radiation [1]. Being mentioned in databases, colour can help in phase identification, however sometimes it is misleading. Even small amount of a substance with strong tinting strength can change sample colour. Colour of powders can be influenced by the moisture. Wet powders are darker and with larger purity than the dry ones. Characterisation of colour ought to be objective, measurement performed with spectrophotometers or colorimeters and colour expressed in such systems as x,y,Y or L,a,b. calculated.

In x, y, Y system, natural co-ordinates i.e. dominant wavelength, brightness and purity can be expressed (Fig.1.): -dominant wavelength determined as a intersection S of line connecting white illuminant W and colour of sample C with chromacity curve-Y is the brightness perpendicular to the x,y plane.- purity being the abscissa WC/WS ratio.

Rheological properties of suspensions have impact on the way of X-Ray measurement of suspensions and pastes and choice of goniometer type. As for suspensions with low viscosity, stirred cuvette is suitable. For the higher viscosity, vertical sample holder and steady sedimentation state can be recommended. [2] Vertical position of the cuvette in the goniometer minimizes the influence of the sedimentation of particles because their motion is parallel to the measured surface. Since the irradiated area is located in the middle of the cuvette and the velocity of the sedimentation is low, the number of particles which enter and leave irradiated volume is constant over a relatively long time. According to the Stokes law, sedimentation time for the spherical particles can be computed

$$\tau = \frac{9a\eta}{2gr^2(\rho_s - \rho_l)}$$

τ time of sedimentation
ρ_s density of solid particles
ρ_l density of liquid
r radius of particle
η dynamic viscozity
g gravitational acceleration
a depth of sedimentation

In such way, times for vertical cuvette where upper edge is 7 mm above radiated area for sedimentation of rutile particles in water and glycerol were computed. Till such time particle concentration in radiated area is not affected by sedimentation. In similar way, times for 1 μ thick layer of the horizontal cuvette is determined. After such time is the layer free of regarded particles.

For suspensions with high viscosity or for pastes, sample holders in vertical position and theta –theta goniometer is recommended. As for no-newtonian suspensions, thixotropy and slip yield value play a role.

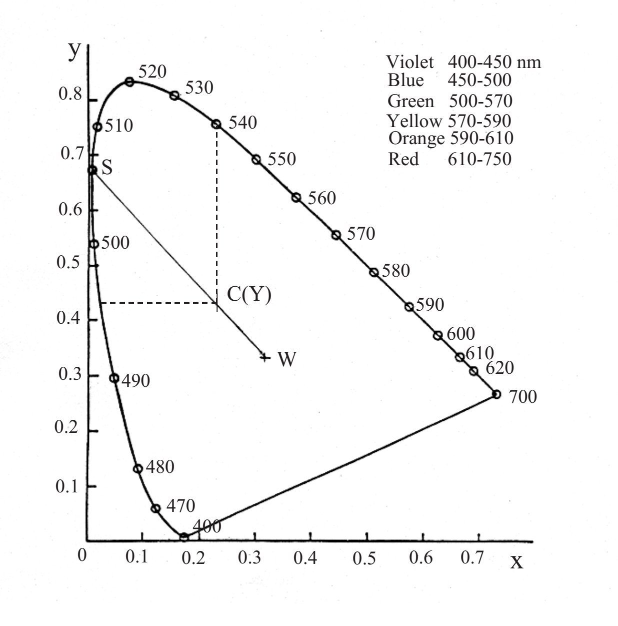

Figure 1. Determination of natural coordinates in x,y,Y colour systém

Table 1. Sedimentation times for depth 7 and 0.001 mm for rutile spherical particles in water and glycerol

Particle radius (10^{-3} mm)	Depth of sedimentation (mm)	Sedimentation time (min) in	
		water	glycerol
0.1	7	1647	Don´t wait for it
1	7	16.5	30530
2.5	7	2.87	4885
10	7	0.16	305
0.1	0.001	0.24	436
1	0.001	0.0024	4.4

References

1. Norm - ČSN 01 1718 Měření barev, Vydavatelství norem Praha , 1990
2. J. Rathouský, G. Schultz-Ekloff, J. Had, A. Zukal: Time resolved in situ X-Ray diffraction study of MCM-41 structure formation from homogeneous enviroment. *Phys. Chem. Chem. Phys*. **1** (1999), 3053-3057