Diffuse Reflectance of Powders

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ABSTRACT THE PROPAGATION OF LIGHT THROUGH POWDERS DIFFERS SIGNIFICANTLY FROM THE PROPAGATION OF LIGHT IN A HOMOGENEOUS MATERIAL, SINCE THE LIGHT SCATTERS OFF POINTS IN ITS PATH. COLORS OF POWDERS ARE RELATED TO THE FINENESS OF THE POWDER, WHEN THE POWDER IS ILLUMINATED WITH WHITE LIGHT. AS FOURIER TRANSFORM INFRARED HAS BECOME MORE COMMON, THE DIFFUSE REFLECTION METHOD HAS BECOME WIDELY USED. DIFFUSE REFLECTANCE IS AN EXCELLENT SAMPLING TOOL FOR POWDERED MATERIALS.

Keywords: powder, diffusive reflectance, absorbance, reflectance

1.INTRODUCTION

Diffuse reflectance was developed to facilitate analysis of materials such as powders [1]. The common characteristic of these materials is their internal inhomogeneities. The propagation of light through such inhomogeneous media differs significantly from the propagation of light in a homogeneous material, since the light scatters off points in its path. The key to the theoretical description of diffuse reflection lies in the description of the propagation of light through inhomogeneous materials [2]. The most widely used model for diffuse reflection is the one developed by Kubelka and Munk [3]. In addition to the fundamentals, the results of the study of the relationship between degree of grinding and the apparent colour of the powder have a wide range of practical applications including: food production, paints, in density and pharmacology, for painting and ceramics, as well as the conservation and restoration of historical artifacts [4,5].

In external reflectance, the energy that penetrates one or more particles is reflected in all directions and this component is called diffuse reflectance [6]. When light shines onto a powder sample, it is reflected in all directions, as depicted in Fig. 1. Some of the light undergoes specular reflection at the powder surface. The remainder of the light is refracted as it enters the powder, where it is scattered due to internal reflection, reflection from the surfaces of other powder grains, or repeated refraction entry into the powder. As the diffuse reflected light is reflected or passes through the powder, it becomes weaker if absorption by the powder occurs.

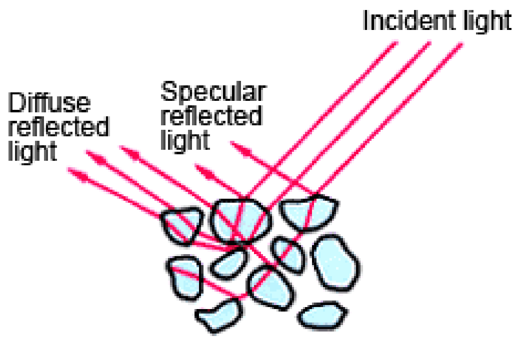

Fig.1. Illustration of diffuse reflectance.

Diffuse reflectance was developed to facilitate analysis of materials such as powders. The common characteristic of such materials is their internal inhomogeneities. The propagation of light through such inhomogeneous media differs significantly from the propagation of light in a homogeneous material, since the light scatters off points in its path. Thus the key to the theoretical description of diffuse reflection lies in the description of the propagation of light through inhomogeneous materials and only approximate descriptions exist.

The most widely used model for diffuse reflection is the one put forward by Kubelka and Munk (K-M). The diffuse reflectance is given as:

$$R = 1 + \frac{k}{s} - \sqrt{\frac{k}{s}\left(2 + \frac{k}{s}\right)}, \quad (1)$$

where *k* is absorption coefficient, *R*- reflection coefficient and *s* – scattering coefficient. This relatively simple form is easily solved for *k*/*s* yielding the familiar K-M transform [7]:

$$\frac{k}{s} = \frac{(1-R)^2}{2R}. \quad (2)$$

This model holds when the particle size is comparable to, or smaller than the wavelength of the incident light, and the diffuse reflection no longer allows to separate the contributions of the reflection, refraction, and diffraction (*i.e.* scattering occurs).The K-M transform of the measured spectroscopic observable is approximately proportional to the absorption coefficient and hence is approximately proportional to the concentration. The scattering coefficient s is, in fact, dominated by particle size and refractive index of the sample. It is not a strong function of the wavelength or the absorption coefficient, so the K-M model considers it a constant. In reality, the scattering coefficient does vary slowly with wavelength. Two constants are needed to describe the reflectance: absorption coefficient *k* scattering coefficient *s*. For $k \to 0$ (no absorption) $R \to 1$, i.e. all light reflected. When $s \to 0$ (no scattering) $R \to 0$, i.e. all light transmitted or absorbed.
The absorbtion in the powder grains depends strongly on the light wavelength. Since *k* depends on the light wavelength, the reflection coefficient *R* depends also. Dependence of the reflection coefficient *R* on wavelength determines the powder color as illustrated in Fig. 2.

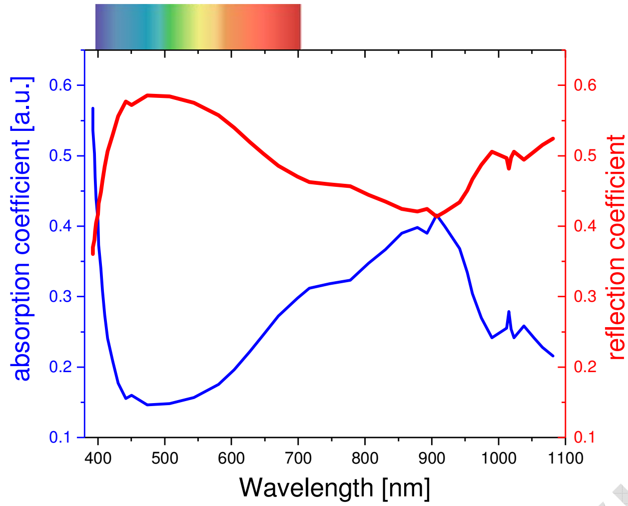

Fig. 2. Absorption and reflection coefficient as a function of wavelength of Copper(II) sulfate.

Light emitted from the powder contains specular reflection light, as well as diffuse reflected light. In order to obtain a more accurate diffuse reflectance spectrum the specular reflection light must be reduced by reducing the particle size. As Fourier transform infrared (FTIR) has become more common, the diffuse reflection method has become widely used. In this paper we present the wave number dependence of the absorbance and reflectance of sugar, corn and wheat [8,9].

2. MATERIAL AND METHODS

Fourier transform infrared (FTIR) is the preferred method of infrared spectroscopy used to obtain an infrared (IR) spectrum of absorption of a solid, liquid or gas. When IR radiation is passed through a sample, some radiation is absorbed by the sample and some is transmitted [10]. The resulting signal at the detector is a spectrum representing a molecular 'fingerprint' of the sample. The FTIR spectrometer uses an interferometer to modulate the wavelength from a broadband infrared source. The FTIR spectra are usually presented as plots of intensity versus wavenumber (in cm^{-1}). Wavenumber is the reciprocal of the wavelength. The intensity can be plotted as the percentage of light transmittance or absorbance at each wavenumber.

In our study the absorbance was obtained by FTIR method by using KBr technique, in the range 250-4000 cm^{-1}. This involves mixing the sample with KBr powder, forming it into a pellet, and measuring the infrared spectrum. It is the basic measurement method for powder samples. However, as FTIR has become more common, the diffuse reflection method has become widely used. The reason for the popularity of this method is the much easier pretreatment than the KBr pellet method. Measurements were performed for sugar, corn and wheat.

3. RESULTS AND DISCUSSION

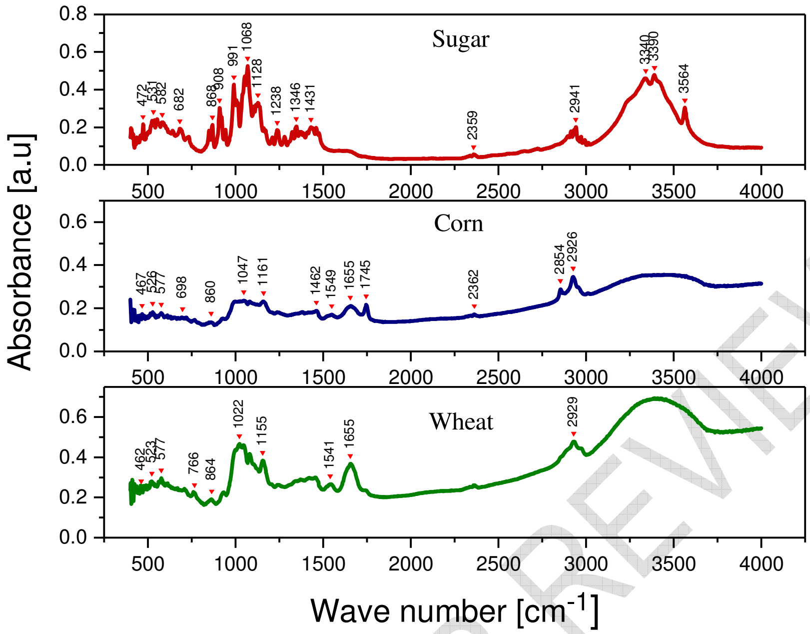

Fig.3. Absorbance versus wave munber of: sugar (red), corn (blue) and wheat (green).

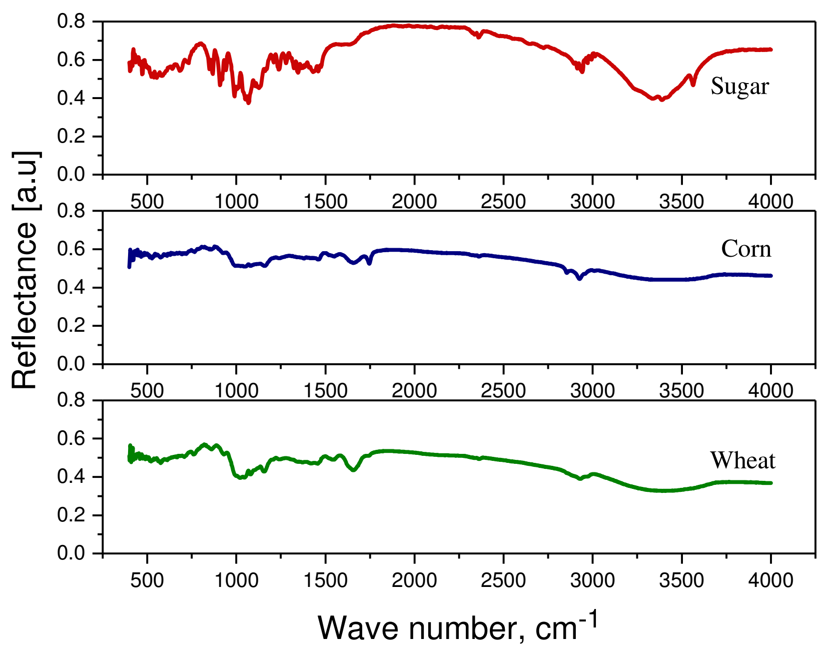

Fig.4. Refectance as a function of wave munber of: sugar (red), corn (blue) and wheat (green).

FTIR spectra for sugar, corn and wheat measured are shown in Fig. 3. The IR spectrum of the sugar contains characteristic groups. From the IR spectrum one may bserve: bands corresponding to wavelengths of 2941 cm^{-1} in sugar, 2926 and 2854 cm^{-1} in corn and 2929 cm^{-1} in wheat (C-H vibration CH_3 and CH_2 groups ; at 1745 and 1655 cm^{-1} in wheat and corn (vibration C = O group); at 1431 and 1462 cm^{-1} for sugar and corn (C-H deformation vibration CH_2 and CH_3 groups); at 1128, 1161 and 1155 cm^{-1} for sugar, corn and wheat, respectively (C-O symmetrical extending ester vibration). A broad band at 3100-3600 cm^{-1} for corn and wheat corresponds to the OH group of molecules. By using expression (1) and the data presented in Fig. 3 we determined reflectance of: sugar, corn and wheat and plotted in Fig.4. As expected, reflectance for all materials is high.

4. CONCLUSION

Reflectance spectra are influenced by a variety of factors due to the different ways in which light scatters, including variations in the refractive index, particle size, ect. Diffusion affects the color since it determines the average path of ligh in the material and therefore to what extent different wavelengths are absorbed. Colors of powders are related to the fineness of the powder, when the powder is illuminated with white light. Particle dimensions affect the color of the powder and color changes occur due to diffuse reflection on powder particles. Reducing the size of the sample particles reduces the contribution of reflection from the surface. Diffuse reflectance is an excellent sampling tool for powdered materials. The technique based on the diffusive reflectance does not require a powder sample to be dispersed in any liquid medium, so the material is not contaminated or consumed.

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