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APPLICATION OF NIR SPECTROSCOPY FOR MONITORING DIFFERENT PARTICLE SIZES OF SUCROSE
PRIMJENA NIR SPEKTROSKOPIJE ZA ODREĐIVANJE RASPODJELE VELIČINE ČESTICA SAHAROZE

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ABSTRACT

Near infrared spectroscopy has become increasingly important non-destructive analytical technique in food science and technology. The aim of this research was to investigate the possibility of using NIR spectroscopy for potential monitoring of the milling process and sorting of *different particle sizes of sucrose. Commercial sucrose was milled using pestle and mortar to obtain different particle sizes. Fractions of different particle sizes ranging from 10 μm to 1000 μm were collected by sieving and monitored i*n the near infra red range from 899 - 1699 nm. Analysis of the spectrums by *principal component analysis (PCA) for sample clustering* revealed that it is possible to monitor different particle sizes of sucrose thus *enabling use of NIR for on-line monitoring of the sucrose milling process.*

***Key words**: NIR, PCA, sucrose, milling.*

REZIME

Blisko infracrvena spektroskopija (NIR) je postala važna ne-razarajuća analitička tehnika u nauci o hrani i tehnologiji. Cilj ovog istraživanja je ispitati mogućnost koriščenja NIR spektroskopije za monitoring procesa mlevenja i sortiranja različitih veličina čestica saharoze. Komercijalna saharoza mlevena je korištenjem tučka i maltera da se dobiju različite veličine čestica. Frakcije različitih veličina čestica u rasponu od 10 μm do 1000 μm dobivene sitenom analizom i praćene su u bliskom infracrvenom opsegu od 899 - 1699 nm. Analiza spektra primenom analize glavnih komponenata (PCA) za grupirane uzorke otkrila je da je moguće pratiti različite veličine čestica saharoze omogučujući korišćenje NIR za on-line monitoring procesa mlevenja saharoze.

Ključne reči: NIR, PCA, saharoza, mlevenje.

INTRODUCTION

One of the main challenges of food science and technology is to obtain precise information about food products that are on a market and their production process. NIR (near infrared) spectroscopy has become increasingly important non-destructive analytical technique in food science and technology which provides online monitoring of production process and gives information of final product. Use of spectroscopy in the NIR region allows a wide range of applications in food chain production, controlling the quality indicators of raw materials, intermediary products and final products (*Ružičkova and Šustova, 2006*) in order to provide a guarantee for consumers (*Damez and Clejron, 2008*). It is well known, however, that NIR spectra contain both chemical information of samples as well as physical information, such as particle size and bulk density (*Pasikatan et al., 2001*).

Most of the previous studies of particle size and its influence on NIR spectra and possible application for in line monitoring of technological process were conducted on pharmaceutical powders. *Liew et al.* (2009) used NIR spectroscopy for in-line quantification of drug and excipients in cohesive powder blends.

They found that this technique may be used to monitor the relative distribution of individual blend components in real time and thus, to asses, the performance of a bin blending for mixing of cohesive multi-component powder blends during development and production. *Saraguca et al.* (2010) used NIR for determination of flow properties of pharmaceutical powders. They showed the potential of NIR spectroscopy in the determination of physical properties affecting the flowability of pharmaceutical powders.

Although there has been a lot of work on pharmaceutical powders, research done on food powders is still not so abundant, probably because of a very complex nature of food powders.

Norgaard et al. (2005) in their work multivariate near-infrared and Raman spectroscopic quantifications of the crystallinity of lactose in whey permeate powder found that both spectroscopic techniques are suitable for determining the lactose crystallinity in whey permeate powder, but NIR was able to predict the crystalline content with a significantly lower prediction error. When implementing analytical technologies on-line in food industry, several new factors need to be investigated including spectrometer performance, sampling device, temperature, mixing, and particle size.

The objective of the presented study is to evaluate the use of NIR spectroscopy for sorting of different particle sizes of sucrose.

MATERIALS AND METHODS

Sucrose sample

Commercial white sucrose (Viro, Virovitica), packed in paper purchased from supermarket was used in this study.

Sample preparation

Commercial sucrose was milled using pestle and mortar to obtain different particle sizes at ambient temperature of 22 °C and 43 % air relative humidity. Fractions of different particle sizes ranging from 10 μm to 1000 μm were collected by sieving for 15 minutes using Analysette 3 PRO laboratory shaker (Fritsch, Germany) and analytical sieves (Fritsch, Germany). After sieving, each fraction was removed from the sieve and placed in a small metal vessel for NIR powder samples and the probe of the NIR instrument was leaned upon the sample with the probe slightly touching the sample. Ten measurements were conducted for each particle size.

NIR measurements

Sucrose NIR spectra were collected in the range of 904-1699 nm using a Control Development, Inc., NIR-128-1.7-

USB/6.25/50µm shown in Fig. 1 with installed Control Development software SPEC 32.

The technique of NIR spectroscopy is based on the electromagnetic absorption at the near-infrared region but the spectral analysis has to be assisted with various chemometric techniques (*Ding & Xu, 1999; Alishahi et al., 2010*). In this case principle component analysis (PCA) was used. PCA analysis is commonly used to identify patterns in experimental data and to express the data based on their similarities and differences.

Pre-processed spectra by Savitsky-Golay "smooth" algorithm for data filtering, which is available by Control Development software Spec32, and first derivation of "smooth" algorithm were recorded in EXCEL format. Mean values of the ten measurements for each particle size were calculated and mean values with according wave lengths were imported to STATISTICA v. 9. software for evaluation.

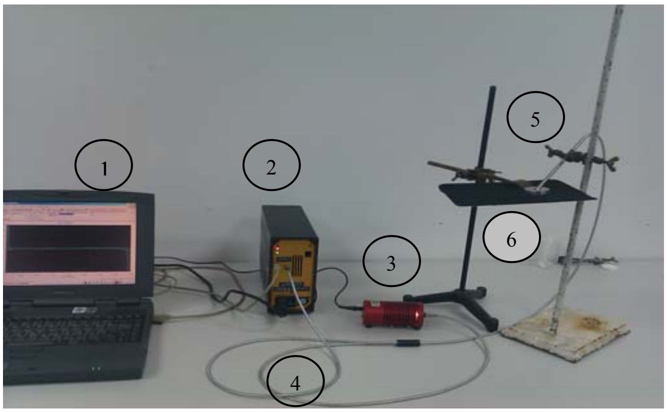

Fig. 1. NIR instrument connected to PC (laptop) (1) Laptop with SPEC 32 software, (2) NIR instrument, (3) source of light, (4) optical cable, (5) measurement probe, (6) sucrose sample in small metal vessel

RESULTS AND DISCUSION

Using NIR process analyzer each spectrum of different particle sizes of sucrose was recorded in EXCEL format. For each particle size ten samples were recorded and their mean value was calculated.

Three typical samples of pre-processed spectra by Savitsky-Golay "smooth" algorithm for data filtering for particle sizes of 250, 500 and 710 µm samples are presented in Fig. 2. In order to perform PCA analysis applied is numerical derivation as presented in Fig. 3.

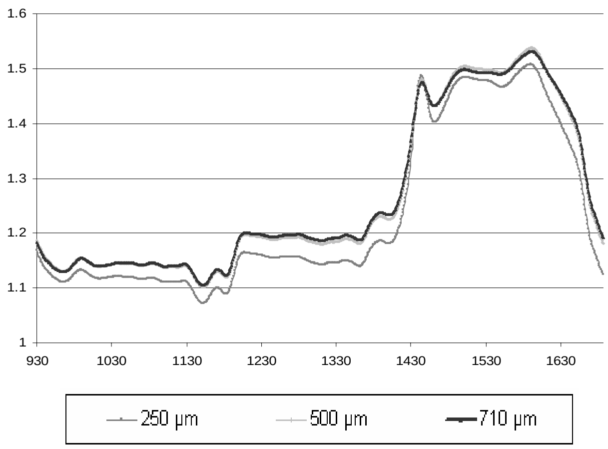

Fig. 2. NIR spectra by Savitsky-Golay "smooth" algorithm for 250, 500 and 710 µm sucrose particle size samples

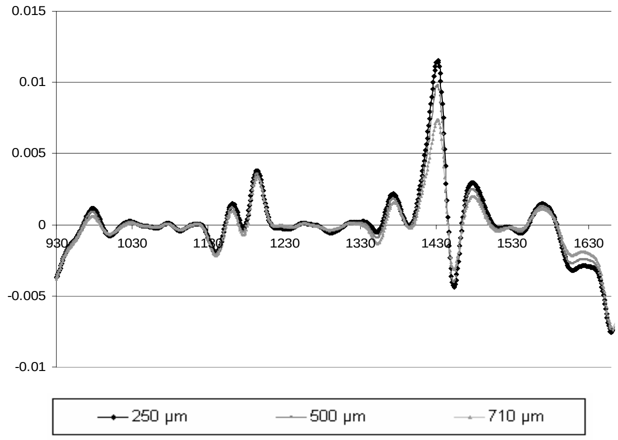

Fig. 3. NIR spectra coresponding to the first derivatives for 250, 500 and 710 µm sucrose particle size samples

Table 1. Eigenvalues of the covariance matrix of NIR spectra.

Value number	Eigenvalue	% Total variance	Cumulative Eigenvalue	Cumulative %
1	216.0318	71.77135	216.0318	71.7714
2	74.9856	24.91217	291.0174	96.6835
3	5.6878	1.88962	296.7052	98.5731
4	3.0558	1.01520	299.7609	99.5883
5	0.7571	0.25152	300.5180	99.8399
6	0.2608	0.08665	300.7788	99.9265
7	0.1647	0.05471	300.9435	99.9812
8	0.0565	0.01877	301.0000	100.0000

In order to apply NIR spectra for sample classification according to particle size principal component analysis (PCA) was applied. Eigenvalues for the data matrix X composed of the ten independent measurements for each particle size were determined first. Eigenvalues were calculated by numerical matrix inversion algorithm provided by STATISTICA v.9 software. Results are presented in Table 1 and graphically as scree plots in Fig. 4. The pronounced affect of the first two eigenvalues was observed and it was also observed by the cumulative effect that 96.7 % of variance was accounted by the first two principal components.

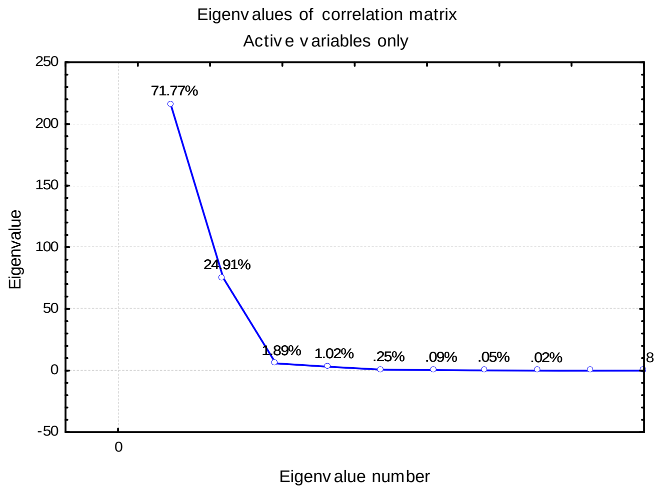

Fig. 4. Scree plots for the NIR spectra of sucrose samples.

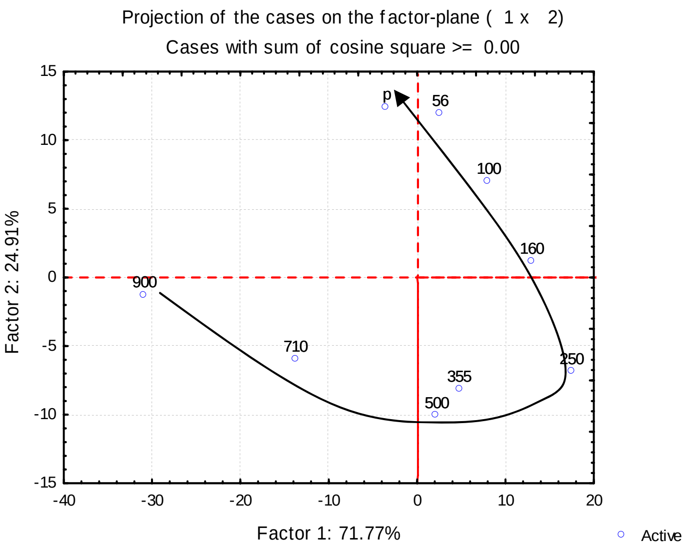

Fig. 5. Classification of different particle sizes of sucrose samples and the milling curve based on NIR spectra in the plane of the first two principal components. The milling curve starts at the particle size of 900 µm up to 56 µm and the underflow fractions are denoted by letter p.

CONCLUSIONS

NIR process analyzer proved to be sufficiently accurate for recognition of different particle sizes of sucrose. Analysis of the spectrums by principal component analysis (PCA) for sample clustering revealed that different particle sizes resulted in different spectrums which opens the possibility of using NIR spectroscopy for in line monitoring of the milling process of sucrose and possibly other food powders with a well defined chemical composition.

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