The application of near infrared spectroscopy (NIR) technique for non-destructive investigation of mixed milk powder products

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Summary

The production of cow's milk in Hungary fluctuates by 15-20 % annualy. Surplus milk is dried into powder and can also be converted to modified milk powders using techniques such as ultra filtration. From approximetely 20.000 tonnes, of all milk powder types, 3.000 tonnes, is converted using ultra filtration technology.

Multivariable near infrared (NIR) calibration was performed on powder mixtures of whole milk, skimmed milk, whey, retenate (protein concentrate) and lactose for rapid fat, protein, lactose, water and ash content determination.

More than 150 samples were prepared and measured in two NIRS labs (Scottish Agriculture College – SAC – Aberdeen and University of Horticulture and Food Science - UHFS – Budapest). The results obtained from the same samples were compared.

The aims of the study were:

1. Rapid quantitative and qualitative determination of mixtures of milk powder products using NIR technique.

2. Comparison of the results achieved in Aberdeen (SAC) and Budapest (UHFS) institutes.

The mass per cent varied between 0.0-2.8% for fat, 0.0-80% for protein, 6.6-100 % for lactose, 0.0-5.0 % for water and 0.0-8.0 % for ash. High correlation coefficients (0.97-0.99) were found for all five components.

Key words: powdered milk, milk protein, lactose, near infrared spectrophotometry, NIR reflectance spectra

Introduction

Multivariate NIR calibration was performed on powder mixtures of whole milk, skimmed milk, whey, retentate and lactose for fat, protein, lactose, water and ash content determination. More than 150 samples were prepared and measured in two NIRS labs (Scottish Agriculture College Aberdeen and UNIVERSITY of Horticulture and Food Science Budapest), the results obtained from the same samples were compared.

So the aims of the study were:

-Rapid quantitative and qualitative determination of mixtures of milk powder products using NIR technique.

-Comparison of the results achieved in Aberdeen (SAC) and in Budapest (UHFS) institutes.

Materials and methods

In the next table (Table 1.) the materials used in the investigations are shown.

Table 1: Composition of the raw materials (%) used
Tablica 1: Sastav upotrebljenih sirovina (%)

Constituens Komponente	Skimmed milk Obrano mlijeko	Whey powder Sirutka u prahu	Protein conc. Proteinski konc.	Lactose Laktoza
Fat Mast	0.9	2.8	0.5	0.0
Protein Protein	36.3	15.3	80.3	0.0
Lactose Laktoza	50.9	73.1	6.6	100.0
Water Voda	4.1	1.4	4.8	0.0
Ash Pepeo	7.8	7.4	7.8	0.0

Four raw materials were used and blended in different ratio for producing the calibration and prediction samples. First 104 samples were prepared. In order to see the influences of the fat content samples with high fat content milk powder were blended was well. So 48 additional samples were prepared.

In Aberdeen a NIR Systems 6500 scanning NIR monochromator, in Budapest a PMC Spectralyzer 1025 scanning type spectrophotometer was used.

For statistical evaluation multivariate analysis was performed using the software the NIR instruments were furnished with. The qualitative evaluation and the drawings were performed using the PQS software.

Results and discussion

In Figure 1. the log(1/R) spectra of the raw materials used in the experiments are presented.

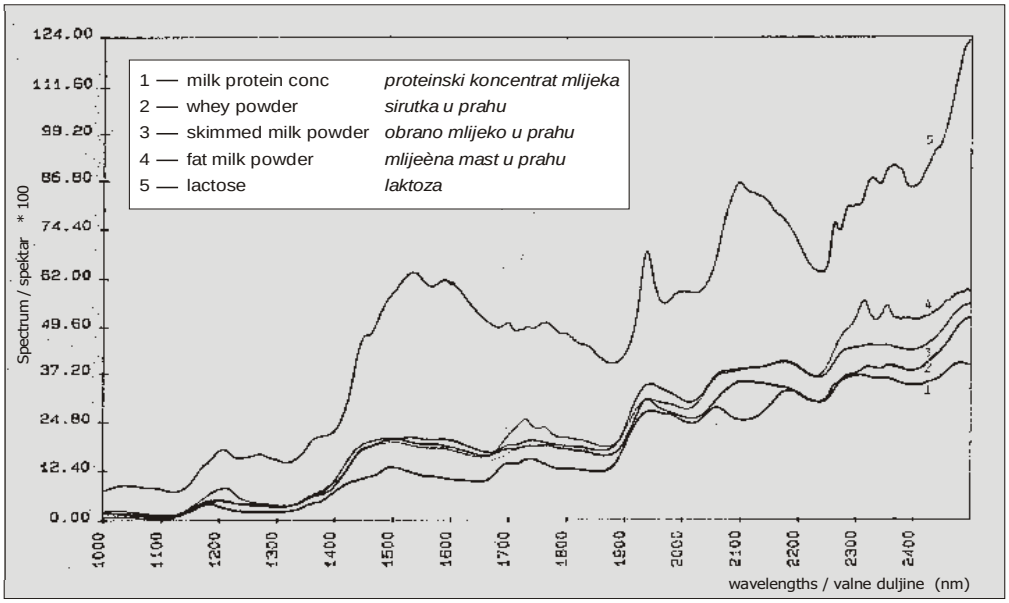

Figure 1: The log (1/R) spectra of the raw materials
Slika 1: Log (1/R) spektar sirovina

The differences between the peaks, show the differences between the concentrate of the constituents.

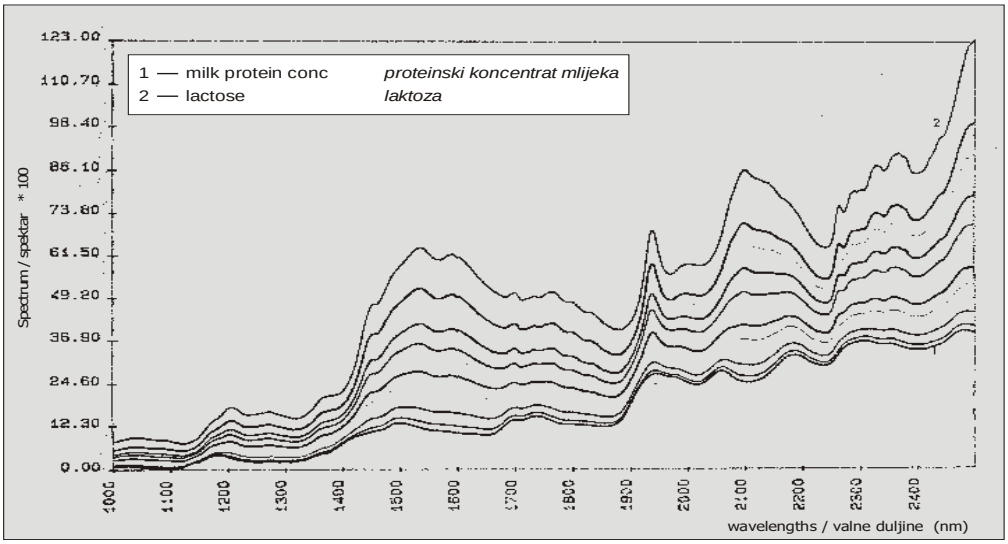

Figure 2: The log (1/R) NIR spectra of the mixtures of protein concentrate and lactose

Slika 2: Log (1/R) NIR spektar smjese proteinskog koncentrata i laktoze

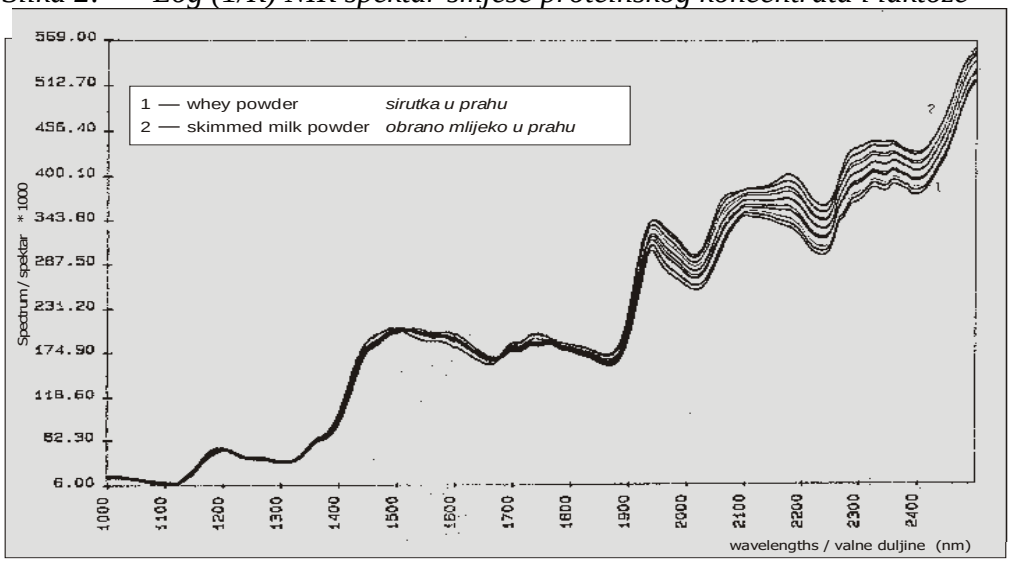

Figure 3: The log (1/R) NIR spectra of the mixtures of skimmed milk and whey powder

Slika 3: Log (1/R) NIR spektar smjese obranog mlijeka i sirutke u prahu

In Fig.2. the log(1/R) spectra of the mixtures of protein concentrate and lactose are shown.

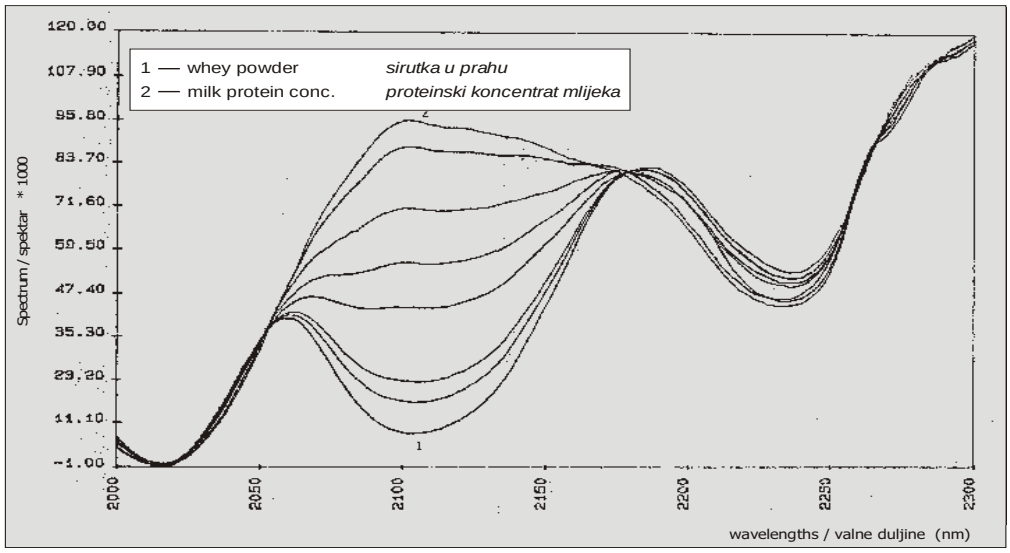

Figure 4: The zoomed log (1/R) NIR spectra of the mixtures protein concentrate and whey powder

Slika 4: Uvećanje log (1/R) NIR spektra za smjesu koncentrata i sirutke u prahu

It's quite a big difference between the spectra of this two products.

Contrary to this figure, in the Fig.3. the NIR spectra of the mixtures of two very similar milk powder products: skimmed milk and whey powder are presented.

Difference can be observed in the 2000-2300 nm wavelength region.

This difference is more perceptible in the zoomed log(1/R) NIR spectra of the mixtures of protein concentrate and whey powder (Fig.4.).

- Curve no.1 is the NIR spectra of the milk protein concentrate in the 2000-2300 nm wavelength region.
- Curve no.2 is the NIR spectra of the whey powder.

At the 2060 nm and 2180 nm we can observe two peaks in the spectra, corresponding to the protein, and at the 2100 nm one peak corresponding to the lactose present in the whey powder.

Statistically evaluating the results, next table (Table 2.) shows the performance data obtained by SAC NIR instrument.

Table 2: The performance data obtained SAC NIR instrument
Tablica 2: Podaci mjerenja dobivenih na SAC NIR instrumentu

Constituents Komponente	Best Wavelenghts (nm) Optimalne valne duljine	Stand. error (SEC) (mass%) Standardna pogreška	Correl. coeff. (R) Koeficijent korelacije	Calibr range (Mass) Baždarno područje
Fat Mast	912 1234 1486 1666 2464	0.125	0.977	0 - 2.8
Protein Protein	762 1774 2146	4.27	0.933	0 - 80.3
Lactose Laktoza	1330 1654 1978	6.79	0.929	6.6 - 100
Water Voda	906 1486 1552 1660 2378	0.161	0.988	0 - 4.8
Ash Pepeo	1150 1378 1642 1846 2092	0.179	0.99	0 - 7.8

At the best wavelengths the correlation coefficients were between 0.93 and 0.99. The values of the standard error for the protein and lactose constituents were relatively high, but the calibration range was wider than at the other constituents.

Looking to the performance data (Table 3.) obtained by UHFI NIR instrument, we can observe the same very good correlation (the values were between 0.98-0.99).

Table 3: The performance data obtained by UHFI NIR instrument

Tablica 3: Podaci mjerenja dobiveni na UHFI NIR instrumentu

Constituents Komponente	Best wavelengths (nm) Optimalna valna duljina	Stand. error (SEC) (mass%) Standardna pogreška	Correl. coeff. (R) Koeficijent korelacije	Calibr. range (mass%) Baždarno područje
Fat Mast	2036 2082 2130 2132 2480	0.128	0.986	0 - 2.8
Protein Protein	1454 1878 1932 2110 2336	1.89	0.997	0 - 80.3
Lactose Laktoza	1442 1460 1464 2144 2284	1.879	0.997	6.6 - 100
Water Voda	1508 1636 1638 2378	0.098	0.998	0 - 4.8
Ash Pepeo	1208 1266 1932 1938 2474	0.257	0.994	0 -7.8

For the protein and lactose the standard error values were higher as well, but lower than in Aberdeen.

In Fig.5 the qualitative evaluation by PQS method is presented.

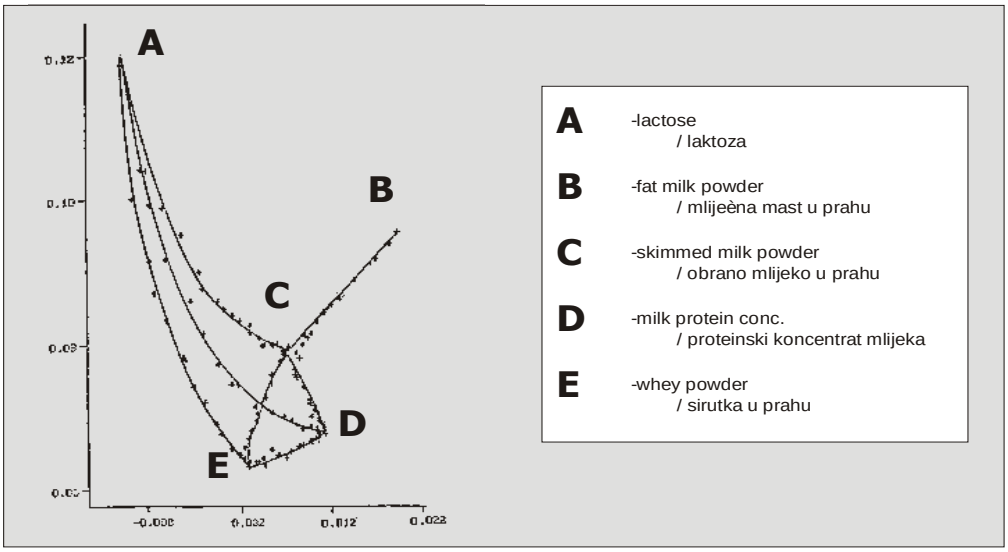

Figure 5: Qalitative evaluation by PQS. Quality points of the milk powder samples, obtained as the gravity points of log (1/R) spectra represented in polar coordinate system from 1000 to 2500 nm

Slika 5: Kvalitativna analiza pomoću PQS. Bodovi kvalitete uzoraka mlijeka u prahu, dobivenih kao ponderirani bodovi log (1/R) spektra i predočeni u polarnom koordinatnom sustavu od 1000 do 2500 nm

The quality points of the milk powder samples, were obtained as the gravity points of log(1/R) NIR spectra represented in polar coordinate system from 1000 to 2500 nm.

In each quality point, all information from the NIR spectra of the investigated sample is concentrated.

In the conclusions is possible to establish:

- Scanning type NIR instruments can be used as multicomponent analyzers for determining the percentages of the constituents in the milk powder mixtures.
- The achievable accuracy for the five constituents is given in table. It is acceptable for industrial practice.
- The PQS qualification system proved to be very useful and simple method without time consuming calibration.

PRIMJENA TEHNIKE BLISKE INFRACRVENE SPEKTROSKOPIJE (NIR) ZA NEDESTRUKTIVNO ISPITIVANJE PROIZVODA OD MLIJEČNOG PRAHA

Sažetak

Proizvodnja kravljeg mlijeka u Mađarskoj u porastu je svake godine prosječno 15-20 %. Suvišak mlijeka najčešće se suši (mlijeko u prahu) ili se pak proizvode modificirani mliječni prahovi metodama poput ultrafiltracije. Od ukupnih 20.000 tona mliječnog praha godišnje se proizvodi 3.000 tona mlijeka u prahu primjenom ultrafiltracije.

Multivarijantna bliska infracrvena spektrofotometrija (NIR) je korištena za brzo određivanje udjela masti, proteina, laktoze, vlage i pepela u mješavinama prahova dobivenih iz svježeg mlijeka, obranog mlijeka, sirutke i proteinskih koncentrata.

Više od 150 uzoraka priređeno je i mjereno u dva NIRS laboratorija (Scottish Agriculture College – SAC – Aberden i University of Horticulture and Food Science – UHFS – Budapest). Komparirani su rezultati dobiveni za iste uzorke.

Svrha rada bila je:

1. *Brzo kvalitativno i kvantitativno određivanje produkata mlijeka u prahu korištenjem NIR tehnike.*
2. *Usporedba rezultata dobivenih u Aberdeenu (SAC) i Budimpešti (UHFS).*

Udio masti varirao je između 0,0 i 2,8 %, udio proteina između 0,0 i 80 %, udio laktoze između 6,6 i 100 %, udio vode između 0,0 i 5,0 % te udio pepela između 0,0 i 8,0 %. Koeficijent korelacije za svih pet komponenti bio je između 0,97 i 0,99.

Ključne riječi: mlijeko u prahu, mliječni proteini, laktoza, bliska infracrvena spektrofotometrija (NIR), NIR spektar

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