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# Research on Real-Time Portable Quality Evaluation System for Raw Milk

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Abstract The goal of this research was to develop a portable system that could be used to evaluate the quality of milk in real time at a raw milk production site. A real-time portable quality evaluation system for raw milk was developed to enable non-destructive quality evaluation of somatic cell count (SCC), fat, protein, lactose, and total solid (TS) in milk samples. A prediction model of SCC, fat, protein, lactose, and TS was constructed using partial least squares (PLS) and 200 milk samples were used to evaluate the prediction performance of the portable quality evaluation system and high performance spectroscopy. Through prediction model development and verification, it was found that the accuracy of high performance spectroscopy was 90% for SSC, 96% for fat, 96% for protein, 91% for lactose, and 97% for TS. In comparison, the accuracy of the portable quality evaluation system was relatively low, at 90% for SSC, 95% for fat, 92% for protein, 89% for lactose, 92% for TS. However, the measurement time for high performance spectroscopy was 10 minutes for 1 sample, while for the portable quality evaluation system it was 6 minutes. This means that the high performance spectroscopy system can measure 48 samples per day (8 hours), while the portable quality evaluation system can measure 80 (8 hours). Therefore, it was found that the portable qual

ity evaluation system enables quick on-site quality evaluation of milk samples.

**Keywords** Visible, Near Infrared Spectroscopy, Portable, Quality Evaluation, Milk

### 1 Introduction

Korea's dairy industry achieved rapid growth within a relatively short period of time compared to other food industries. Annual milk production in 2013 was 201 tons, and it is expected to be 220 tons in 2014. Also, average annual consumption of milk has been continuously increasing, in the range of 2-3% per year (KDBFA, 2013). However, while both the production and consumption of milk are continuously rising, the domestic self-sufficiency rate of milk has been consistently dropping because of a constant increase in the availability of cheap and high quality imported milk, causing an imbalance between supply and demand of milk (Jang, 2001). For this reason, supply control and the production of high quality milk are required to enhance the international competitiveness of Korean milk.

High quality milk means fresh milk that is rich in flavor and nutrients such as protein and fat, while being low in bacteria and somatic cells. An increase in the number of somatic cells in milk causes a reduction in the amount of milk and the amount of nutrients in milk such as fat, lactose, protein, and casein, and an increase in the amount of natrium and chlorine in milk, affecting the quality of raw milk. When this occurs, dairy farmers are hit by significant economic losses, as the value of their milk drops substantially. Somatic cell count in raw milk is also used for diagnosis of mastitis in cows. Therefore, controlling the quality of raw milk through a quick on-site measurement of the somatic cell count is required.

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Currently, quality evaluations of raw milk rely on sample evaluation, instead of total evaluation. Sample evaluation is carried out on the milk samples created by mixing milk samples extracted from many cows. This makes it difficult to selectively remove raw milk with low quality. As low quality milk is mixed with other milk, it reduces the overall quality of the mixed milk. In addition, to perform a quality evaluation using near-infrared spectroscopy, the milk samples collected at farms must be taken to a laboratory, and although the evaluation process is automated, the evaluation time is still quite lengthy. For this reason, a real-time portable evaluation system that can evaluate the quality factors of raw milk in real time at the milking facility is needed to produce high quality milk.

Therefore, this research developed a portable real-time quality evaluation system that enables the automatic pre-processing of samples including milk sample supply, reagent injection, temperature control and mixing of samples, and non-destructive measurement of SSC, fat, protein, lactose, and TS, utilizing the optical properties of milk, and conducted a comparative analysis with a high-performance spectrometer of prediction performance and measurement time.

#### 2 Materials and methods

# 2.1 Milk sample

200 milk samples were collected in cooperation with Korea Food Research Institute to include milk of grades 1 through 5 produced in Gyeonggi-do, Gangwon-do, and Jellanam-do between October and December, 2010. After the milk samples were collected, they were treated with preservatives to minimize the change in contents because of the surrounding environment. Then, the somatic cell count and components composition were evaluated at Seoul Milk Ansan Branch using the standardized raw milk component analyzer, Combifoss.

Table 1 Grade of milk by somatic cell count

Grades	SCC
1	less than 200,000
2	200,000-350,000
3	350,000-500,000
4	500,000-750,000
5	more than 750,000

# 2.2 Reagent

Raw milk component analysis using visible and near-infrared spectroscopy produces a good prediction model when a milk sample that is intact is used, however more outstanding correlations can be observed when a reagent is added to the sample for the spectroscopy conducted on-site in real time (Choi et al., 2008a).

Therefore, Resazurin that is used for dye reduction test for measuring microorganism (Choi et al.(2008a)) was used as a reagent that influences somatic cell count. The dye reduction test measures the ability of microorganisms in milk. Milk components are used for oxidation and deliver the electrons created during the oxidation to oxygen or dye molecules to reduce them. Reduction time is inversely proportional to the number of microorganisms in milk. When the somatic cell count increases, reduction time quickens (Lee, 2005).

# 2.3 Raw milk portable quality evaluation system development

The raw milk portable quality evaluation system was built in a case sized 42x25x30cm (width\*length\*hight) as shown in Fig.1 for easy transport and moving, for on-site, real-time raw milk quality evaluation.



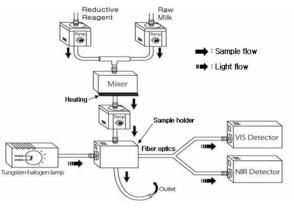


Fig. 1 Real time portable quality evaluation system and schematic diagram of portable milk quality evaluation system.

Spectroscopy involves a light source, spectrometer, and optical fiber. For the light source, a tungsten-halogen lamp that can be used stably at 360 to 2000 nm was used. The spectrometer used visible spectra (350-1000nm) and near-infrared spectra (850-1700nm) to reflect the spectral characteristics of milk. For optical fiber, 300  $\mu$ m Singe fiber was used.

If the user presses the power button of the system, Windows starts and a program to operate the portable quality evaluation system runs on the LCD screen. The user can evaluate the quality of milk on-site by touching the screen. Lithium-ion battery cells were used for the system to make it portable so that it can measure the quality of raw milk on-site in real-time. It can be recharged with input voltage of AC 90~250V and continuously perform quality evaluation for 6 consecutive hours on site. The system consists of power supply for on-site evaluation, a preprocessor for raw milk and Resazurin solution supply, mixing and temperature control, light source, sample holder, and detection unit for quality evaluation, and embedded device to control the whole system.

I/O interface is connected with the embedded module via USB connection, enabling high speed communication. It has a maximum of 8 channels analog input, 12 channels digital input/output, and counter and analog output of 1 channel, and its 14-Bit resolution enables 48k sampling. The embedded module uses a Windows-based micro-controller and has USB, TCP/IP, and Serial port, providing easy communication with outside. The flow pump of the sample supply unit that controls raw milk and reagent provides raw milk and reagent to mixing container by ON/OFF on the I/O interface. The thermoelement of the temperature control unit that controls the temperature of mixed samples maintains a steady temperature of mixed samples by digital output of I/O interface that receives the temperature of the thermoresistor. Also, the spectra of measured samples are transformed into absorbance (log(1/R)) and stored in the embedded module through RS-232C communication. Spectroscopy for quality evaluation consists of light source, sample set, detection unit, and light probe. Specifications are described in Table 2.

Table 2 Specifications of I/O interface, embedded module and portable quality evaluation system

Items	Specifications			
	Function	Analog in/out, Digital in/out, Counter-timer		
I/O interface module	Resolution	14 bit(48 kS/s)		
	Input range	-10 ~ +10		
Embedded controller	Microprocessor	VIA Luke CoreFusion TM processor		
	I/O Connectors	USB, RS-232C, TCP/IP		
	OS	Microsoft windows xp		
Light source	Lamp	Tungsten-halogen		
	Spectral range	360 ~ 2000 nm		
	Power	12 VDC / 6.5 watts		
Fiber-optics	Fiber type	Singe fiber, 300 µm diameter		
Sample cell & holder	Flow cell	1.5 mm pass length		
	Wavelength	350 ~ 1000 nm		
VIS Detector	Data interval	0.18 nm		
	Detector	Linear CCD array 3648 pixels		
	Wavelength	850 ~ 1700 nm		
NIR Detector	Data interval	1.7 nm		
_	Detector	InGaAs linear array 512 pixels		

A system control algorithm was developed to control the whole system including the sample supply device, sample mixing device, temperature control device, and spectrum measurement device. The control program in Fig. 2 was developed using Labview version 8.2 (National Instrument, USA), a system-design platform that enables the easy development of measurement and control programs.

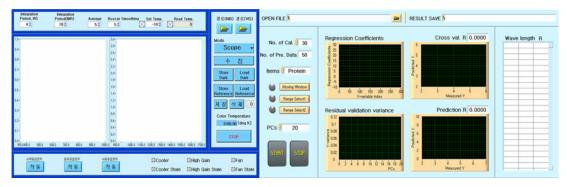


Fig. 2 Control program of real time portable quality evaluation system of milk.

When the user turns on the real-time portable raw milk quality evaluation system, each part of the system is provided with power by the power supply and an 'Initializing' message appears on the screen, along with a message saying '10 minutes is required to preheat the light source and temperature control device.' After preheating the system, select the supplying milk button and supplying reagent button to provide raw milk and reagent to mixing cell at a speed of 6.1 mL/min, which is the optimum blending speed of raw milk and reagent, controlled by speed pump. Supplied milk and reagent are mixed by a rotor at a speed of 5,020 rpm for 5 minutes (Choi et al., 2008b). The temperature control unit was developed to maintain the temperature of 40  $^{\circ}\mathrm{C}$ , the ideal temperature for reaction, by turing on/off the thermoelement according to the PID control algorithm whose input is the temperature of thermoresistor attached to the mixed cell. (Pradova et al., 2001). The mixed sample reacts for 5 minutes, the ideal reacting time for raw milk and the reagent (Kim et al., 2008). After the reaction, the user can provide the mixed sample to Flow cell using the sample supply button. After supplying the sample, the sample supply pump stops for spectrum measurement and the spectrum of the sample is measured. Measurement of the sample can be performed using the data collection button of control program, and optical properties signals detected by the visible/near-infrared spectrometer are transformed to electric signals through the interface module of the spectrometer to be transformed to absorbance in the embedded system control device and stored. After the spectrum measurement, the sample supply pump is operated by the sample supply button to discharge the sample. After the measurement of one sample is completed, water is supplied to the raw milk supply line to clean the silicon tube, mixing cell, and sample holder so that measurement of the next sample can proceed in the same way. The measured spectrum data can be printed using the printer connected with the embedded device through TCP/IP, serial, or USB port.

#### 2.4 Spectrum measurement

In order to develop a raw milk components prediction model, the whole spectra acquired using the portable system and high performance spectroscopy was separated into a calibration set of 50% and a verification set of 50%. Components were arranged in order of size and sequentially separated into a calibration set and verification set to have a calibration set and validation set with a similar size and range. As you can see in Fig.3, after heating up the preserved milk to 40°C, Resazurin test was carried out on the raw milk sample for 5 minutes to acquire spectra. The portable system used visible and near infrared spectrometer (Fig. 4 left) to measure spectra at 350 to 1000 nm and at 850 to 1700 nm, while high-performance spectroscopy (NIRS 6500, Foss, Denmark) (Fig. 4 right) measured spectra at 400 to 1700 nm.

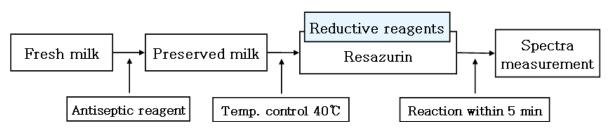


Fig. 3 Schematic of experiment condition for spectra measurements.



Fig. 4 Portable VIS spectrometer(left), NIR spectrometer(middle), and laboratory VIS/NIR spectrometer(right).

# 2.5 Quality evaluation algorithm development

The prediction model split the spectra of raw milk samples into 100 calibration sets and 100 verification sets, and used partial least squares (PLS), which is appropriate for analysis of samples of various chemical components, to analyze the spectra of raw milk samples and correlations of somatic cell count (SCC), fat, protein, lactose, and total solids. For PLS analysis, a self-developed program was used for the portable quality evaluation system, while a commercial program, Unscrambler (Camo, Norway) was used for high performance spectroscopy. In order to develop a prediction model, multiplicative scatter correction (MSC), a mathematical treatment for correction of raw spectrum and light scattering was used. Also, cross validation was used for the PLS model to increase the reliability of the developed model and set the maximum number of factors to 20. In addition, correlation coefficients (R) and standard errors of calibration (SEC) for each factor were compared to develop the optimum model. The prediction performance of the developed model was evaluated using correlation coefficient (R) of prediction and standard error of prediction (SEP).

# 3 Results and discussion

# 3.1 Milk sample analysis

The results of the analysis of the milk samples using

Combifoss are shown in Table 3. Somatic Cell Count was between 15,000 and 1,322,000, revealing that the samples include milk of grades 1 through 5. Fat content was found to be 2.47-6.40%, protein content was 2.50-4.25%, lactose content was 3.91-5.23%, and total solids content was 10.59-15.81%.

Table 3 Chemical properties of milk tested

(N = 200)

				,
	Avg.	Max.	Min.	Std.
SCC (ea)	344,954	1,322,000	15,000	282,775
Fat (%)	4.02	6.40	2.47	0.81
Protein (%)	3.31	4.25	2.50	0.44
Lactose (%)	4.71	5.23	3.91	0.42
TS (%)	12.71	15.81	10.59	0.71

# 3.2 Spectrum measurement

Reflectance spectra were acquired through visible spectroscopy using fiber optic probe and high performance near-infrared spectroscopy. As shown in Fig. 5, similar patterns were observed at 350 to 1000nm and 850 to 1700nm on the reflectance spectra aquired using both forms of spectroscopy, but overall absorbance was lower at the reflectance spectra of visible spectroscopy. The reason is considered to be the reduced intensity of the radiation of visible spectroscopy.

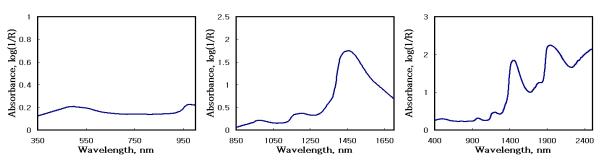


Fig. 5 A typical reflectance spectra of milk sample using portable VIS spectrometer(left), portable NIR spectrometer(middle), and laboratory VIS/NIR spectrometer(right).

### 3.3 Prediction model development

The result of developing a prediction model of milk components using a portable quality evaluation system and high performance spectroscopy is shown in Table 4. In the case of development of prediction model using high performance spectroscopy, SCC was observed to have correlations at 400 to 600 nm, correlation coefficient was 0.93, and error was 8,375 cells. Fat was found to have correlations at 1,300 to 1,500 nm, correlation coefficient was 0.99, and error was 0.08%. Protein was observed to have correlations at 1,400 to 1,700 nm, correlation coefficient was 0.98, and error was 0.10%. Lactose was observed to have correlations at 1,200 to 1,700 nm, correlation coefficient was 0.93, and error was 0.16%. TS (total solids) was found to have correlations at 1,300 to 1,500 nm and correlation coefficient was 0.99, and error was

0.09%.

As a result of developing a prediction model using the portable raw milk quality evaluation system, the correlation coefficient of the prediction model was relatively lower than high performance spectroscopy. SCC showed correlations at 400 to 700 nm, and correlation coefficient and error were 0.91 and 10,961 cells, respectively. Fat was observed to have correlations at 1,200 to 1,500nm, and correlation coefficient and error were 0.98 and 0.11%. Protein was shown to have correlations at 1,400 to 1,600 nm, and correlation coefficient and error were 0.95 and 0.19%, respectively. Lactose was observed to have correlations at 1,300 to 1,600 nm, and correlation coefficient and error were 0.90 and 0.18%, respectively. TS was observed to have correlations at 1,300 to 1,500 nm, and correlation coefficient and error were 0.96 and 0.23%, respectively.

Table 4 PLS result of calibration for each spectrum measurement

		Wavelength	Factor	R	SEC
SCC(ea)	Laboratory	400~600	8	0.93	8,375
	Portable	400~700	8	0.91	10,961
Fat(%)	Laboratory	1,300~1,500	7	0.99	0.08
	Portable	1,200~1,500	8	0.98	0.11
Protein(%)	Laboratory	1,400~1,700	8	0.98	0.10
	Portable	1,400~1,600	7	0.95	0.19
Lactose (%)	Laboratory	1,200~1,700	9	0.93	0.16
	Portable	1,300~1,600	10	0.90	0.18
TS(%) -	Laboratory	1,300~1,500	11	0.99	0.09
	Portable	1,300~1,500	10	0.96	0.23

# 3.4 Performance evaluation of portable quality evaluation system of raw milk

The result of the on-site evaluation of the portable raw milk quality evaluation system is shown in Table 5. Preheating time of high performance spectroscopy is 30-40 minutes, while that of the portable milk quality evaluation system is 10 minutes. This reveals that the portable milk quality evaluation system required less preheating time to make on-site valuation easier.

In terms of portability, it is impossible to carry a high performance spectroscopy system to use at a raw milk production site. This type of system can only be used in a laboratory and in a stable state, while the portable quality evaluation system is easy to carry and move, making an on-site real-time quality evaluation of milk components possible.

In terms of the preprocessing of raw milk sample and reagent, if high performance spectroscopy is used, users have to manually preheat the milk and mix it with reagent and supply it using constant temperature water bath and micro-pipette. However, the portable system was developed to automatically carry out supply, mix, and temperature control of raw milk and reagent.

Regarding the prediction performance of raw milk components, the accuracy of high performance spectroscopy is 90% for SCC, 96% for fat, 96% for protein, 91% for lactose, and 97% for TS, while the accuracy of the portable quality evaluation system is 90% for SCC, 95% for fat, 92% for protein, 89% for lactose, and 92% for

TS. This means that the accuracy of the portable quality evaluation system is comparatively lower. Measurement errors of high performance spectroscopy were 10,863 for somatic cell count, 0.38% for fat content, 0.16% for protein, 0.19% for lactose, and 0.27% for TS, while errors of the portable quality evaluation system were 13,111 cells for SCC, 0.45% for fat, 0.23% for protein, 0.33% for lactose, and 0.38% for TS. So, the measurement errors of the portable quality evaluation system were relatively higher.

As for the measurement time, high performance spectroscopy takes 10 minutes for 1 milk sample, while the portable quality evaluation system takes 6 minutes. This means that the high performance spectroscopy can handle 48 samples a day (8 hours) while the portable quality eval-

uation system can handle 80 samples a day. Therefore, it was judged that the portable quality evaluation system enables faster quality evaluation. High performance spectroscopy presents higher prediction performance than the portable quality evaluation system, However, milk samples are required to be carried to the laboratory, and the measurement time is longer. Also, in consideration of the high price of high performance spectroscopy, it is thought to be difficult to use it on a production site. Meanwhile, the portable quality evaluation system requires 6 minutes to evaluate one sample, and real-time quick evaluation is possible without the need to move the milk samples. This enables milk producers to evaluate the quality of milk on site, and thus can be expected to contribute to high-quality milk production.

Table 5 Comparison of portable quality evaluation system and laboratory system

	Portable quality evaluation system	High performance spectroscopy
Preheating time	10 minutes	30-40 minutes
Portability	portable	not portable
Preprocess	automatic	manual
Additional equipment	milk sample, reagent container	milk sample, reagent container a constant-temperature water bath, micro pipette, PC
Evaluation method	transmittance spectra	reflectance spectra
Accuracy	SCC: 90%	SCC: 90%
	Fat : 95%	Fat : 96%
	Protein: 92%	Protein : 96%
	Lactose: 86%	Lactose: 91%
	TS: 92%	TS : 97%
	SCC(ea): 13,111	SCC(ea): 10,863 cells
	Fat : 0.45%	Fat : 0.38%
Measurement error	Protein : 0.23%	Protein: 0.16%
	Lactose: 0.33%	Lactose: 0.19%
	TS : 0.38%	TS : 0.27%
Measurement time	6minutes/sample	10minutes/sample
Measurement method	transmittance spectra	reflectance spectra
Price of system	approximately 40 million Korean won	approximately 130 million Korean won

# 4 Summary and Conclusions

In this research, a device was designed to evaluate the quality of milk in real-time, and a trial product was built. Using the quality evaluation system, this research acquired the reflectance spectra of milk and developed a prediction model of raw milk components. The quality evaluation

system was composed to control the whole system, including sample supply device, sample mix device, temperature control device, and spectrum measurement device. The system predicted correlations of somatic cell count, fat, protein, lactose, and total solids through PLS analysis algorithm. It showed relatively lower milk components prediction accuracy, but its advantages in terms of port-

ability and mobility shows its potential for real-time on-site quality evaluation of milk components.

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