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Comparative Evaluation of Total Solids, Freezing Point and Specific Gravity of Raw Milk Using an Ultrasonic Milk Analyzer Versus Standard Methods

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ABSTRACT

Forty five composite raw milk samples were each subdivided into five portions and then combined with 8, 4 or 0% water, and 2 or 5% whole milk powder (WMP). Ten samples randomly selected from each subsample were comparatively analyzed for their solid content using an ultrasonic milk analyzer (UMA) versus the standard sand-pan oven dry method. Similarly, fifteen samples from each subsample were analyzed for freezing point using the UMA versus a cryoscope. All milk subsamples were also analyzed for specific gravity using the UMA and a lactometer versus the standard gravimetric method. The solid content of the respective milk subsamples averaged 12.61, 12.93, 13.84, 15.31 and 17.52 % while those produced by the UMA averaged 12.81, 13.41, 13.69, 16.02 and 18.82 %. On average, the UMA readings provided a difference of 0.64 % higher ($P<0.05$) milk solids than the standard method. Mean freezing points of the milk subsamples analyzed by the two methods were -0.512, -0.531, -0.542, -0.651 and -0.799°C for the UMA and -0.493, -0.508, -0.549, -0.662 and -0.854°C for the cryoscope, respectively. The mean specific gravity determined by the three analysis methods increased ($P<0.05$) from 1.0265 to 1.0278, 1.0292, 1.0340 and 1.0407 for the five respective milk subsamples. The UMA as well as the lactometer consistently provided higher ($P<0.05$) specific gravity values than the standard oven dry method for all milk subsamples. However, on average, a difference of only 0.0014 specific gravity was observed between the UMA and the standard method. Contrary to this, a greater difference ($P<0.05$) of 0.0043 specific gravity was evident between the lactometer and the standard method. It was therefore considered advisable that the UMA instruments be regularly calibrated with the local raw milk standard. It was also recommended that the accuracy of lactometers be evaluated and their specific gravity readings be adjusted accordingly.

Key words: raw milk, total solids, freezing point, specific gravity, ultrasonic milk analyzer

INTRODUCTION

Milk solids content and/or specific gravity are still used by milk-collecting centers and certain milk factories in developing countries, including Thailand, as the primary criteria for judging possible water adulteration as well as for the payment of raw milk. However, for most well-developed milk manufacturers, the raw milk freezing point has been the criterion of choice for determining water adulteration (Harding, 1995). Determination of milk solids and specific gravity using the standard methods is time consuming. Although the determination of the freezing point

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using an automatic cryoscope is relatively fast, the instrument is rather expensive. Consequently, these analysis techniques are usually not adopted by small to medium-scale milk collecting centers and manufacturers in most developing countries. Under such circumstances, the lactometric method is normally employed for analyzing milk specific gravity and total solids due to its low cost and ease of use. However, it is generally accepted that the lactometer produces only an indirect estimate of the specific gravity and milk solids content. According to Bradley *et al*. (1992), the lactometer used should not provide a specific gravity reading for raw milk that differs by more than 0.0001 from that of the standard reference. On the other hand, the Ultrasonic Milk Analyzer (UMA) is an alternative instrument that has recently gained popularity among milk-collecting centers and certain milk manufacturers in Thailand for determining the milk composition as well as an estimation of its freezing point. UMAs are designed for fast and cost-effective analysis of raw milk. The unit depends on an ultrasound wave to analyze certain milk components. Estimation of the freezing point of the milk sample is provided via a built-in simulation equation. According to Eon Trading (2001), the instrument can provide readings for milk density and the percent of added water within a range of ± 0.0005 g/cm^3 and ± 5%, respectively. The objective of this study was to comparatively evaluate the total solids content, freezing point and specific gravity of raw milk using an ultrasonic milk analyzer and a lactometer versus the standard method.

MATERIALS AND METHODS

Forty five composite raw milk samples were collected from the Kasetsart University Dairy Center. After thorough mixing, each milk sample was subdivided into five portions and then combined with 8, 4 or 0% water, and 2 or 5% whole milk powder (WMP). Ten samples randomly selected from each raw milk subsample were analyzed for their solid content using the UMA (Ekomilk-M) and the standard sand-pan oven dry gravimetric method (Bradley *et al*., 1992). Similarly, 15 samples randomly selected from each milk subsample were analyzed for their freezing point using the UMA and a manual cryoscope (Gerber Instrument). All milk subsamples were again comparatively analyzed for specific gravity using the UMA, lactometer and standard gravimetric method. The Ekomilk-M instrument was pre-standardized by the manufacturer and the analysis was conducted following the procedures outlined by Eon Trading (2001). The cryoscope was also pre-standardized and the analysis was conducted according to Case *et al*. (1985). The lactometer was similarly pre-standardized by the manufacturer and the analysis was conducted according to Bradley *et al*. (1992). The standard method involved a weight-over-volume gravimetric method using a double decimal pre-standardized digital scale with 500 g maximum weight and a 250 ml volumetric flask. All analyses were carried out in duplicate. Analysis of variance was conducted as a 2 × 5 and 3 × 5 factorial randomized complete block experimental design (RCBD). Least square analysis was used to compare the differences among means (Cody and Smith, 1997).

RESULTS

Analysis using the standard oven dry gravimetric method revealed that the total solids content for the 8, 4 or 0% water, and 2 or 5% WMP subsamples averaged 12.612, 12.929, 13.839, 15.310 and 17.521 %, respectively (Table 1). The UMA produced relatively greater ($P<0.05$) total solids values with averages of 12.812, 13.414, 13.691, 16.018 and 18.822% for the five respective subsamples. The UMA provided higher ($P<0.05$) total solids reading (average 14.95 ± 2.94%) than that of the standard method (average 14.30 ± 2.40%). Least square means of the freezing points in the composition-modified milk samples

analyzed by the UMA versus the cryoscope are shown in Table 2. The freezing points significantly reduced (P<0.05) from a mean of -0.502 to -0.520, -0.546, -0.657 and -0.827, respectively for the five milk subsamples. On average, the UMA recorded a higher (P<0.05) freezing point than that of the cryoscope (-0.607 versus -0.613 °C). The least square means for the specific gravity of the raw milk subsamples analyzed by the UMA versus the lactometer and gravimetric methods are summarized in Table 3. The least squares means for the specific gravity of the five modified milk subsamples determined by the three analysis methods increased (P<0.05) from 1.0265 to

Table 1 Least square means ± SD of total solids (%) in modified raw milk analyzed by Ultrasonic Milk Analyzer versus oven dry method.

Samples	n	UMA	Oven dry	LSMs ± SD
8% water	10	12.812 ± 1.864 ^{g}	12.612 ± 1.468 ^{g}	12.712 ± 1.666 ^{q}
4% water	10	13.414 ± 1.963 ^{f}	12.929 ± 1.469 ^{g}	13.172 ± 1.716 ^{p}
Raw milk	10	13.691 ± 1.799 ^{e}	13.839 ± 1.295 ^{e}	13.765 ± 1.547 ^{o}
2% WMP	8	16.018 ± 1.997 ^{c}	15.310 ± 1.684 ^{d}	15.664 ± 1.841 ^{n}
5% WMP	10	18.822 ± 1.839 ^{a}	17.521 ± 1.376 ^{b}	18.172 ± 1.608 ^{m}
LSMs ± SD		14.946 ± 2.937 ^{x}	14.302 ± 2.398 ^{y}	14.624 ± 2.686

^{abcdefg} LSMs within the main and sub treatment effects with different superscripts are different ($p<0.05$)

^{mnopq} LSMs within the same column with different superscripts are different ($p<0.05$)

^{xy} LSMs within the same row with different superscripts are different ($p<0.05$)

Table 2 Least square means ± SD of freezing point (ʃC) in modified raw milk analyzed by Ultrasonic Milk Analyzer versus cryoscope.

Samples	n	UMA	Cryoscope	LSMs ± SD
8% water	15	-0.512 ± 0.0055 ^{a}	-0.493 ± 0.0114 ^{a}	-0.502 ± 0.0067 ^{m}
4% water	15	-0.531 ± 0.0048 ^{b}	-0.508 ± 0.0108 ^{a}	-0.520 ± 0.0069 ^{n}
Raw milk	15	-0.542 ± 0.0111 ^{c}	-0.549 ± 0.0093 ^{c}	-0.546 ± 0.0092 ^{o}
2% WMP	15	-0.651 ± 0.0311 ^{d}	-0.662 ± 0.0174 ^{d}	-0.657 ± 0.0391 ^{p}
5% WMP	15	-0.799 ± 0.0602 ^{e}	-0.854 ± 0.0175 ^{f}	-0.827 ± 0.0322 ^{q}
LSMs ± SD		-0.607 ± 0.113 ^{x}	-0.613 ± 0.136 ^{y}	-0.610 ± 0.1276

^{abcdef} LSMs within the main and sub treatment effects with different superscripts are different ($p<0.05$)

^{mnopq} LSMs within the same column with different superscripts are different ($p<0.05$)

^{xy} LSMs within the same row with different superscripts are different ($p<0.05$)

Table 3 Least square means ± SD of specific gravity of raw milk analyzed by UMA versus lactometer and the gravimetric method.

Samples	n	UMA	Lactometer	Gravimetry	LSMs ± SD
8% water	45	1.0260 ± .0005 ^{j}	1.0298 ± .0005 ^{j}	1.0238 ± .0014 ^{r}	1.0265 ± .0008 ^{q}
4% water	45	1.0272 ± .0005 ^{i}	1.0309 ± .0006 ^{i}	1.0253 ± .0019 ^{k}	1.0278 ± .0009 ^{p}
Raw milk	45	1.0284 ± .0005 ^{i}	1.0319 ± .0006 ^{i}	1.0272 ± .0026 ^{i}	1.0292 ± .0025 ^{o}
2% WMP	44	1.0332 ± .0008 ^{e}	1.0360 ± .0008 ^{e}	1.0329 ± .0051^{e}	1.0340 ± .0022 ^{n}
5% WMP	43	1.0407 ± .0014 ^{b}	1.0423 ± .0008 ^{b}	1.0389 ± .0039 ^{c}	1.0407 ± .0020 ^{m}
LSMs ± SD		1.0311 ± .0064 ^{y}	1.0342 ± .0046 ^{x}	1.0297 ± .0079 ^{z}	1.0342 ± .0046

^{abcdefghijkr} LSMs within the main and sub treatment effects with different superscripts are different ($p<0.05$)

^{mnopq} LSMs within the same column with different superscripts are different ($p<0.05$)

^{xyz} Means within the same row with different superscripts are different ($p<0.05$)

1.0278, 1.0292, 1.0340 and 1.0407 for the respectively for 8, 4 or 0% water, and 2 or 5% WMP. Of the three analysis methods, the specific gravity estimate for each milk sample from the UMA and from the lactometer were slightly, but consistently, higher than those from the standard method. The differences in specific gravity values between the UMA and the standard method were 0.0022, 0.0019, 0.0012, 0.0003 and 0.0018, respectively for the 8, 4 or 0% water, and 2 or 5% WMP. On the other hand, the differences in the specific gravity values between the lactometer and the standard method were 0.0060, 0.0056, 0.0037, 0.0031 and 0.0034 for the five respective milk subsamples. With the exception of the differences between the UMA versus the standard analysis for the 0% water and the 2% WMP sub-samples, the remaining differences in specific gravity for the rest of treatments were statistically significant ($P< 0.05$).

DISCUSSION

With the exception of raw milk, the UMA consistently yielded higher total solids content values compared to the standard method for the rest of the composition-modified raw milk subsamples (Figure 1). On average, the total solids content of the milk subsamples estimated by the UMA was about 0.644 units (14.946 versus 14.302 %, respectively) or 4.50 % more than that estimated by the standard method. It was thus anticipated that the freezing points produced by the UMA should be lower than those of the standard cryscopic analysis. However, on average, the UMA yielded a slightly higher freezing point than the standard technique (-0.607 versus -0.613 °C). It was evident that while there was no difference in the freezing point readings between the two methods for raw milk, the UMA produced lower prediction values for diluted raw milk samples but higher values for raw milk with high solids content (Figure 2). The specific gravity values for the five milk subsamples given by the UMA were relatively close to but higher than the specific gravity values from the standard method. On average, a difference of 0.0014 units between the two methods (1.0311 versus 1.0297, respectively) was found. This difference was 0.14 % more than the average specific gravity value from the standard method. However, when compared across the range of subsamples from a

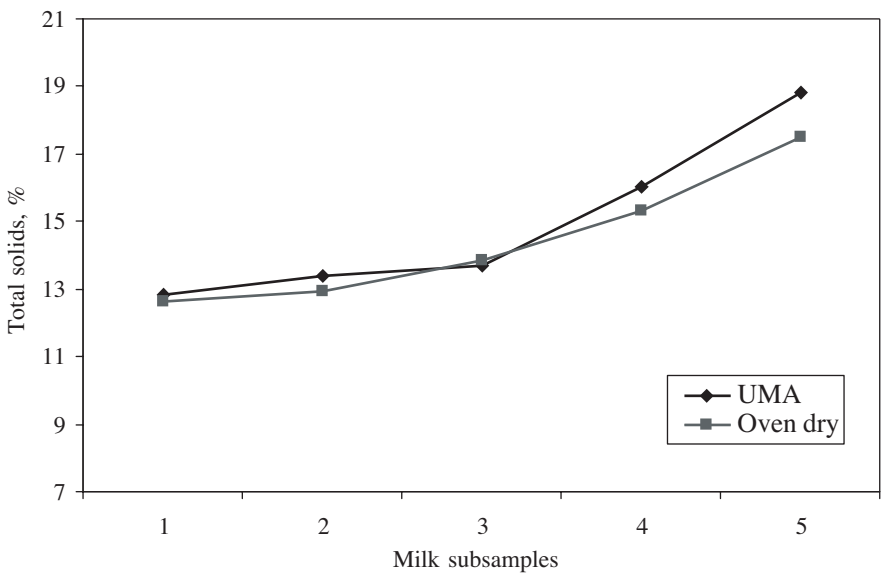

Figure 1 Changing pattern of total solids.

low to high solids content, slightly larger but significant differences in the specific gravity were evident between the two methods for the 8 and 4% water and for the 5% WMP subsamples (Figure 3). These deviations could be rectified by having the instrument regularly calibrated (Weber Scientific, 2003). Figure 4 illustrates the linear relationships between estimates from the UMA and the lactometer compared to the standard gravimetric analysis method. The linear equation to convert UMA (y) to a standard reading (x) is x = 1.0418y – 0.0446. It was interesting to note that the normal acceptable reading range for specific gravity by the UMA was stipulated to be between 1.0260 to 1.0330 (Eon Trading, 2001). In view of the fact that the specific gravity values for the 5%

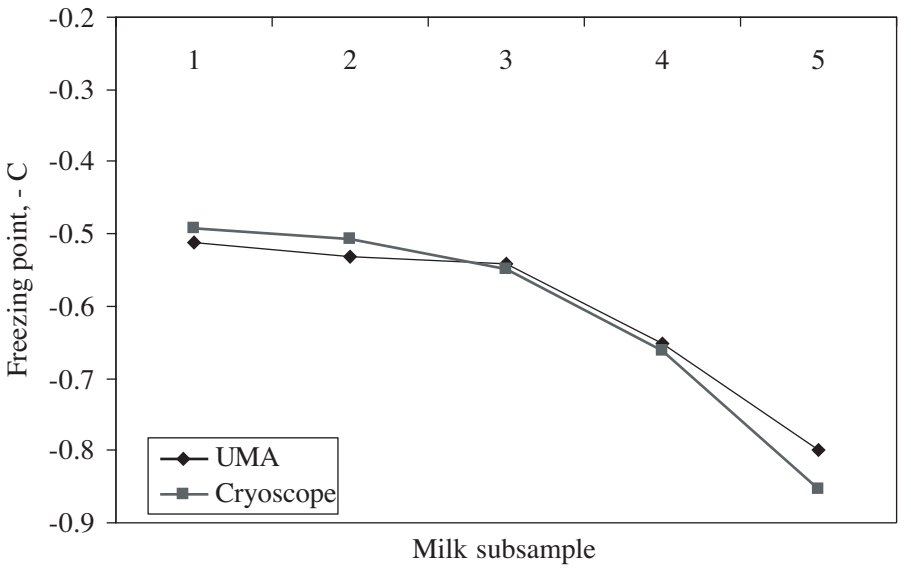

Figure 2 Changing pattern of freezing point.

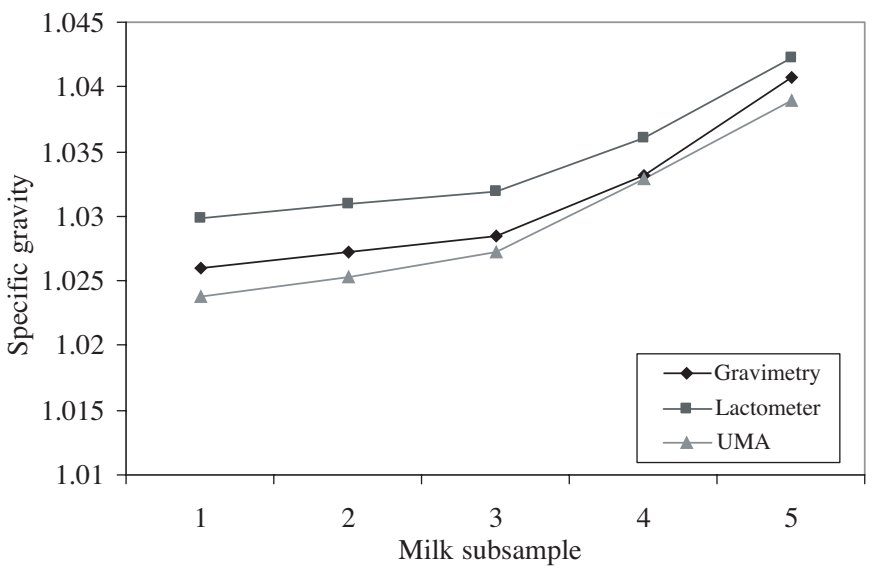

Figure 3 Changing pattern of specific gravity.

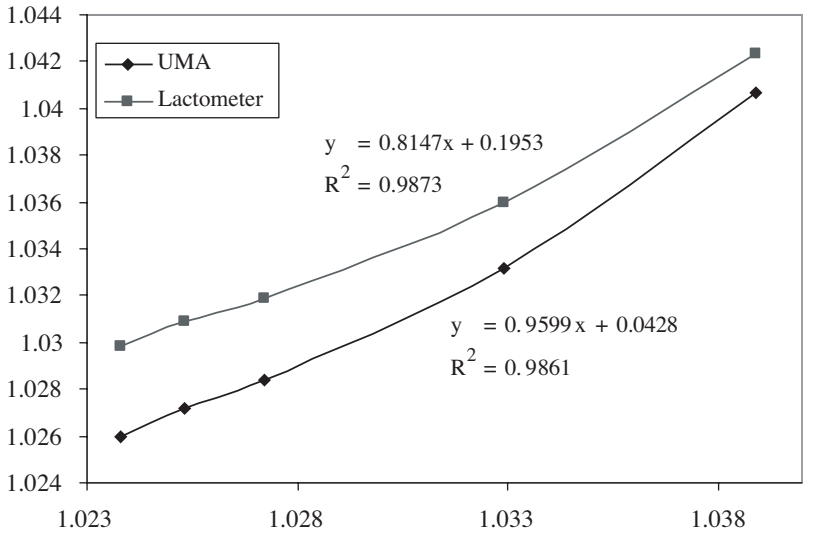

Figure 4 Specific gravity readings from UMA and lactometer (y) versus gravimetric method (x).

WMP subsamples were still well represented by a linear relationship (Figure 4), the acceptable range for specific gravity readings by the UMA could be extended to at least 1.040 instead of 1.0330 stipulated by Eon Trading (2001).

A significant difference ($P<0.05$) for the specific gravity readings from the lactometer versus the standard method was evident across the composition-modified milk subsamples (1.0297 versus 1.0342). The difference of 0.0043 units represents a 0.42 % increase over the readings by the standard method. The fact that the specific gravity readings from the lactometer are consistently lower than those from the standard method provides evidence that the lactometer used in this experiment had not been properly calibrated by the manufacturer. According to Bradley *et al.* (1992), any lactometer producing a deviation of over 0.0001 from that of the reference standard should be discarded. Due to its low cost and ease of use, this type of lactometer is widely used in many milk collecting centers in Thailand. For the lactometer used in this experiment, as shown in Figure 4, the linear equation to convert the lactometer (y) reading to a standard reading (x) is $x = 1.2275y - 0.2397$.

CONCLUSION

UMA instruments should be regularly calibrated with the local raw milk standard. For more precision and convenience to users, the UMA manufacturer should provide re-adjustable built-in simulation equations for each parameter in the micro-processing unit of the instrument. It was also considered advisable that individual lactometers be evaluated prior to purchase. For those already in use, their specific gravity readings should be monitored and adjusted accordingly.

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