A comparison of butter texture measurements with the AP 4/2 penetrometer and TA.XT. Plus texture analyzer

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ABSTRACT
The main aim of this study was to compare the values of selected textural attributes of commercial butter measured with the AP 4/2 cone penetrometer (VEB Feinmess, Germany) and TA.XT Plus texture analyzer (Stable Micro Systems, United Kingdom). Before the analysis, butter samples were stored at a temperature of 8.0 ± 0.5°C for 7 days. The samples were tempered in a water bath in the Binder KB 115 cooled incubator (10.0 ± 0.5°C) for 24 h before the measurements to stabilize their temperature. A total of 18 butter samples with 82% fat content were analyzed. This study demonstrated that butter produced in winter can differ in textural parameters. The results of the measurements conducted with all instruments and probes were highly correlated, and the correlation coefficient reached $r = 0.7–0.9$ at $p < 0.05$. The regression curves well quantified the relationships between the experimental data generated by two measuring systems: firmness measured with the wire cutter probe (WCP) and the P/5 cylinder probe (P/5CP) in the TA.XT Plus texture analyzer (70.49%), and firmness measured with the AP 4/2 cone probe (AP 4/2 CP) and P/5CP (TA.XT Plus) (79.96%). The results of this study indicated that the values of the textural properties of butter measured with different analysis systems can be compared. Penetration tests conducted with the use of AP 4/2 CP or P/5CP (TA.XT Plus) can be rapid and easy to perform, and they can be effectively replaced shear tests.

INTRODUCTION
The textural properties of butter, namely its structure and consistency, are determined by many interrelated parameters such as the concentration, size, shape and distribution of structural elements: fat crystals, fat globules, air bubbles, and droplets of the aqueous phase.[1–7] Butter is one of the most complex edible fats because it contains more than 400 fatty acids. The type of fatty acids in triacylglycerols directly affects crystallization and, in consequence, the structure and physical properties of the end product, such as melting and solidification temperatures and the content of the solid phase at specific temperatures which are highly correlated with the firmness and spreadability of butter.[3–5,8–18]

The texture of fat spreads is the main qualitative attribute that is evaluated in both instrumental and sensory analyses. Firmness and spreadability are the key sensory attributes of butter that are evaluated in consumer acceptability tests.[3,19,20,21] Sensory analyses of textural attributes support comprehensive characterization of food products, including their mechanical, geometric and surface parameters that are perceived by somatosensory (mechanical), sensory, auditory receptors and photoreceptors.[19–25]

Various methods have been developed for evaluating the firmness of edible fats. The textural parameters of edible fats are determined in instrumental and sensory analyses that are highly correlated.[14,26–28] Instrumental methods have a certain advantage over sensory analyses because
they are inexpensive, rapid and objective. They are also easy to standardize, and they are applied under strictly defined and controlled conditions. At present, the textural parameters of butter are determined in various computer-assisted instrumental tests, including the penetration test, shear test, texture profile analysis (TPA) and the back extrusion test.[2,29–36]

The penetration test is simple and cheap, which makes it the most popular instrumental method for evaluating butter texture.[37–39] In this approach, a cylinder, needle, spherical or cone probe is introduced mechanically into the sample to a specified depth at constant speed and force. The probe is selected based on the anticipated range of firmness values or the type of the analyzed product. In general, harder samples require probes with a smaller diameter or a smaller cone angle.[10,29,30,40] Cone penetrometers are widely applied to assess stress-strain properties which are a measure of a product’s consistency and softness.[30,36,39] This method is recommended by the American Oil Chemists’ Society (1989).[41] In this approach, the sample is penetrated by a metal cone with a known mass and geometry. Cone tip resistance increases with depth, and different products are characterized by a specific penetration depth. In a conventional penetration test involving a cone probe, the cone is positioned directly above the surface of the sample, and penetration occurs under the probe’s dead weight. The distance traveled by the probe during a specific time interval (for example, 5 seconds) is a characteristic parameter that describes the sample’s firmness (Table 1).[10,28,36,39,40,42–44]

A texture profile analysis (TPA) is probably the simplest and the most commonly applied instrumental method of food texture analysis. During a TPA, the sample is compressed twice by a flat probe with a diameter larger than the sample’s diameter. Strain, stress and the force required to compress a sample are registered during the test, and the sample is usually compressed to 50% of its original height. A TPA mimics the action of the human jaw.[10,30,45] The test is fast, and it simultaneously generates information about the sample’s main textural parameters, such as firmness, adhesiveness, springiness, cohesiveness, elasticity, gumminess and chewability.[24,25,46] An instrumental TPA well complements sensory analyses, and it produces reliable information about a product’s textural parameters regardless of the sensory panelists’ abilities and preferences.

A shear test measures biting force and the sample’s firmness, shear strength and shear force. The sample is cut to a certain depth using a blade with specific geometric parameters, and its strain (a combination of compressive force, shear force and rupture force) is measured to determine the sample’s cross-sectional characteristics, i.e. the maximum shear force. The higher the maximum shear force, the harder the sample.[23,47]

The back extrusion test is also widely applied to measure the firmness, consistency and spread-ability of butter. A sample is placed in a cylinder with a fixed diameter. The test probe is a flat extrusion plate that is introduced into the cylinder, which causes the product to move upward through the space between the disc and the cylinder’s walls.[10]

Instrumental methods of textural analysis are increasingly applied in the food processing industry and in specialist research institutes. Their main advantages include rapid and simultaneous acquisition of information about various textural attributes and reliable results that are not influenced by the researchers’ emotional state. Instrumental methods are useful in analyses where a large number of samples have to be rapidly tested, and in research studies aiming to develop new and innovative products.[24,27,48,49]

The aim of the present study was to compare the results of instrumental penetration tests (AP4/2 penetrometer, TA.XT. Plus texture analyzer) and a shear test (TA.XT. Plus texture analyzer) in an analysis of the textural properties of commercial butter, and to identify the correlations between the evaluated textural attributes.
Table 1. Parameters of the compared instrumental methods for analyzing the textural attributes of butter samples.

| Device   | Probe                        | Measured parameter | Definition                                                                 |
|----------|------------------------------|--------------------|---------------------------------------------------------------------------|
| AP 4/2   | Cone probe (CP) (H-2520), cone mass = 102.5 g | Penetration [\(\text{\textdegree}p\)] | Firmness was expressed by the penetration depth achieved in 5 seconds, where \(\text{\textdegree}p = .1\) mm |
| Device | Probe | Measured parameter | Definition |
|--------|-------|--------------------|------------|
| TA.XT. Plus | Wire cutter probe (WCP), wire diameter – 0.3 mm | Maximum shear force \( f_{\text{max}} \) | to achieve a penetration depth of 18 mm |
| TA.XT. Plus | P/5 cylinder probe (P/5CP), cylinder diameter – 5.0 mm | Maximum penetration force \( f_{\text{max}} \) | required to achieve a penetration depth of 12 mm |

Firmness \([\text{N}]\)

- Maximum penetration force \( f_{\text{max}} \) required to penetrate the sample to a given depth

Work of penetration \([\text{N}\cdot\text{s}]\)

- The softness of the sample (within the registered range of positive loads) is determined by the work required to penetrate the sample |

Resistance to probe withdrawal/Adhesiveness \([\text{N}\cdot\text{s}]\)

- Work required to withdraw the probe from the sample (within the registered range of negative loads)
MATERIALS AND METHODS

Sample preparation

The experimental material comprised butter with 82% milk fat content. All butters were produced by the continuous method with the use of the applicable production technology, and the characteristic fat composition of winter milk was taken into consideration in the production process.

Butter was produced by different manufacturers, and it was purchased in original packaging in retail outlets in Olsztyn. The products were transported to the laboratory and stored at a temperature of 8 ± 0.5°C for 7 days to stabilize their temperature and promote the formation of a stable crystal structure in butter fat according to ISO 16305:2005 guidelines. Samples for texture analyses were cut out from butter sticks. They were tempered in a water bath in the Binder KB 115 cooled incubator (Tuttlingen, Germany) at 10 ± 0.5°C for 24 h before the analysis. Depending on the test, texture measurements were performed in three and six replicates. A total of 18 butter samples were analyzed and labeled as A, B, C, D, E, F, G, H, I, J, K, L, M, N, O, P, R and S.

Analysis of textural parameters

Textural attributes were analyzed with the AP/42 cone penetrometer (VEB Feinmess, Dresden, Germany) and the TA.XT. Plus texture analyzer (Stable Micro Systems, United Kingdom).

Penetration test with the AP 4/2 cone penetrometer

The penetration test involved a cone penetrometer composed of a hollow brass cone with a 90° tip angle and a stainless steel probe with a mass of 102.5 g (H-2520). The test was conducted according to the procedure described by Staniewski et al. (2006) and Hayakawa et al. (1982) with some modifications. Penetration depth under gravity was measured during 5 seconds. Penetration depth was read directly from the instrument on a scale of 0–400 °p, and it was expressed in penetration degrees [°p], where 1 °p was equivalent to a vertical distance of 0.1 mm traveled by the penetrometer cone in the butter sample (Table 1). Measurements were conducted in six replicates, and mean values were calculated for each sample.

Analysis of textural parameters with the TA.XT. Plus texture analyzer

The remaining textural attributes were determined with the TA.XT. Plus texture analyzer (Stable Micro Systems). A shear test was performed with a wire cutter probe (A/BC) with 0.3 mm wire diameter and the HDP/90 heavy-duty platform. Cubical butter samples with 25 mm side length were used in the test. Butter samples were cut out with a suitably profiled cubical steel puncture probe that was used in the shear test. Penetration depth was 18 mm, the applied load was 0.490 N, and probe speed was 0.5 mm/s. Butter firmness was determined as the maximum shear force [N] required to cut the sample (Table 1). The test was conducted at a temperature of 10 ± 0.5°C, and firmness was measured instrumentally according to ISO 16305: 2005 guidelines (ISO, 2005), where firmness is determined as the average load during probe displacement of 8–16 mm. The results of three measurements were averaged, and a shear test curve is presented in Figure 1.

Butter firmness was also determined during a point penetration test involving a 5 mm cylinder probe (P/5, Stable Micro Systems). Penetration depth was 12 mm, the applied load was 0.049 N, and probe speed was 2.0 mm/s (Table 1). The obtained curves were used to determine firmness as the maximum penetration force [N], work of penetration [N∙s] – as the work required to penetrate the sample to a given depth (within the registered range of positive loads) which is indicative of sample softness, and resistance to probe withdrawal (adhesiveness) [N∙s] – as the work required to separate the probe from the sample (within the registered range of negative loads) (Figure 2) Each sample was tested in six replicates.
Statistical analysis

The results of the measurements were used to calculate arithmetic means and standard deviation [s]. Pearson’s r was determined in a linear correlation analysis (Table 2), and regression curves were plotted to determine the relationships between the results of instrumental texture analyses (Figure 3, Figure 4, Figure 5) with the use of Statistica v. 13.0 software.
| Method | AP 4/2 (CP) | TA.XT. Plus (WCP) | TA.XT. Plus (P/SCP) |
|--------|-------------|-------------------|---------------------|
| Butter producer | 1 Penetration [cm] | 2 Firmness [N] | 3 Firmness [N] | 4 Work of penetration [N·s] | 5 Resistance to probe withdrawal [N·s] |
| A      | 59.50 ±1.87 | 3.67 ±0.10 | 8.96 ±0.39 | 46.45 ±6.00 | −10.90 ±0.88 |
| B      | 58.50 ±4.14 | 3.47 ±0.02 | 9.03 ±0.30 | 42.26 ±7.61 | −8.25 ±0.92 |
| C      | 54.17 ±0.16 | 3.82 ±0.10 | 9.54 ±1.07 | 47.94 ±8.60 | −7.36 ±0.67 |
| D      | 59.83 ±1.94 | 2.61 ±0.11 | 7.34 ±0.40 | 32.37 ±5.43 | −9.60 ±1.30 |
| E      | 56.33 ±2.50 | 3.02 ±0.01 | 8.03 ±0.66 | 38.26 ±7.52 | −9.57 ±1.52 |
| F      | 43.83 ±2.48 | 4.05 ±0.41 | 12.03 ±0.42 | 56.23 ±4.46 | −8.08 ±0.80 |
| G      | 58.67 ±2.16 | 3.66 ±0.00 | 9.21 ±0.39 | 49.68 ±7.75 | −9.81 ±0.82 |
| H      | 74.67 ±2.16 | 1.69 ±0.02 | 5.10 ±0.11 | 25.44 ±2.02 | −10.89 ±0.33 |
| I      | 42.00 ±2.61 | 4.30 ±0.08 | 11.79 ±0.57 | 54.90 ±8.76 | −8.41 ±0.61 |
| J      | 41.33 ±1.51 | 4.08 ±0.22 | 11.08 ±0.60 | 52.15 ±9.27 | −9.73 ±1.40 |
| K      | 57.50 ±2.07 | 2.84 ±0.10 | 7.59 ±0.37 | 34.16 ±7.62 | −7.47 ±1.57 |
| L      | 40.67 ±4.50 | 4.72 ±0.46 | 13.39 ±0.78 | 55.05 ±8.46 | −14.51 ±1.89 |
| M      | 52.00 ±3.10 | 3.22 ±0.11 | 9.05 ±0.49 | 29.96 ±5.18 | −9.10 ±1.13 |
| N      | 48.50 ±2.43 | 3.21 ±0.04 | 9.19 ±0.59 | 40.04 ±17.33 | −11.77 ±1.09 |
| O      | 57.17 ±2.99 | 2.70 ±0.09 | 7.33 ±0.23 | 33.80 ±5.94 | −8.89 ±1.03 |
| P      | 49.17 ±2.64 | 3.22 ±0.26 | 9.10 ±0.12 | 38.13 ±6.20 | −11.74 ±0.33 |
| R      | 45.50 ±1.52 | 3.25 ±0.05 | 10.40 ±0.41 | 55.55 ±12.88 | −10.03 ±0.91 |
| S      | 44.17 ±1.83 | 3.65 ±0.10 | 10.93 ±0.36 | 26.00 ±12.04 | −12.87 ±1.49 |
| ᴿ      | 52.42 ±2.48 | 3.40 ±0.13 | 9.39 ±0.46 | 42.13 ±5.80 | −9.94 ±1.04 |

r = linear correlation coefficient

- 1–2: 0.7587 *
- 1–3: 0.8396 *
- 2–3: 0.8942 *
- 3–4: 0.6285 *
- 3–5: −0.2084 *
- 4–5: 0.0650

Mean ± standard deviation (SD)

* - differences are significant at p < 0.05
Figure 3. Regression curve of the textural parameters measured with the AP 4/2 cone probe and the wire cutter probe (WCP) in the TA.XT. Plus texture analyzer.

Figure 4. Regression curve of the textural parameters measured with the AP 4/2 cone probe and the P/S cylinder probe (P/SCP) in the TA.XT. Plus texture analyzer.
Figure 5. Regression curve of the textural parameters measured with the wire cutter probe (WCP) (TA.XT. Plus) and the P/S cylinder probe (P/SCP) (TA.XT. Plus).

RESULTS AND DISCUSSION

Penetration test involving the AP 4/2 cone penetrometer and firmness test involving the TA.XT. Plus texture analyzer

The firmness of butter samples was measured at 10°C, which is similar to the temperature of butter after it is removed from the refrigerator. In the penetration test conducted with the AP 4/2 cone penetrometer (CP), firmer samples were characterized by lower penetration depth and lower values of \( P \). In the penetration test conducted with the TA.XT. Plus texture analyzer with the P/5 cylinder probe (P/5CP) and in the shear test involving a wire cutter probe (WCP), firmer samples were characterized by higher values of the maximum penetration force and the maximum shear force [N], respectively. The results of instrumental texture analyses and the correlation coefficients of the investigated butter samples are presented in Table 2. The conducted tests and the applied probes produced different firmness values. According to Laia et al. (2000)\(^{[50]}\) samples with a similar solid phase content at a given temperature can differ in firmness. These variations can be attributed to seasonal changes in the composition and properties of milk fat, i.e. the crystallization behavior of milk fat which is determined by processing temperature during cream ripening in butter production. Storage conditions also play an important role.\(^{[5,16,17,51–54]}\) Ronholt et al. (2012)\(^{[16]}\) and Tondhoosh et al. (2016)\(^{[17]}\) observed that the ripening of slowly cooled cream significantly increased butter firmness (due to the formation of a denser crystal network), whereas the ripening of rapidly cooled cream considerably decreased butter brittleness. Staniewski\(^{[4]}\) reported that butter firmness is much higher in winter when milk fat contains lower concentrations of monoenoic and polyenoic fatty acids that indirectly influence crystallization. In a study by Tondhoosh et al.\(^{[17]}\) an increase in the fat content of cream (from 40% to 45%), an increase in ripening time (from 3 h to 5 h) at an average temperature of 18°C, and a decrease in churning temperature (from 12°C to 10°C) led to the production of softer butter with improved spreadability. The above process effectively decreased butter firmness, in particular in winter. In the present study, firmness values were significantly highest in sample L and lowest in sample H, in all
tests. According to Staniewski (2005), the proportion of milk fat globules with a thick membrane in a crystalline state and a liquid triacylglycerol core should be increased during cream ripening in winter to decrease butter firmness and improve the rheological properties of butter.\textsuperscript{[1,3,13,50,55,56]}

The regression curves presented in Figures 3, 4, 5 indicate that all of the examined attributes were bound by linear relationships. The linear regression equation produced a regression line that the best quantified the relationships between the analyzed data. The firmness values measured with WCP and P/5CP were characterized by the highest positive value of the correlation coefficient (r = 0.8942), and they were most accurately predicted (79.96%) by the regression model (R^2 = 0.7996) estimated by the correlation equation \( \gamma = 2.5563x + 0.7021 \) (Figure 5). The firmness values measured with AP4/2 CP and P/5CP (TA.XT. Plus) in the penetration test were bound by a highly significant negative correlation (r = -0.8396), and they were relatively well predicted (70.49%) by the regression model (R^2 = 0.7049) estimated by the correlation equation \( \gamma = -0.1878x + 19.2365 \) (Figure 4). The scatterplot (Figure 3) revealed a highly significant linear correlation (r = -0.7587) between penetration measured with AP4/2 CP and firmness measured with WCP (TA.XT. Plus). The goodness of fit of the regression model estimated by the correlation equation \( \gamma = -0.0594x + 6.5112 \) was only 58%.

It should be noted that the textural attributes measured with all devices in all instrumental analyses of sample firmness were highly (p < .05) correlated at \( r = 0.7–0.9 \). The scatterplots of firmness values measured with WCP and P/5CP (TA.XT. Plus texture analyzer) and firmness values measured with AP4/2 CP and P/5CP (TA.XT. Plus) in the penetration test revealed that regression curves well fit the experimental data at R^2 = 0.7–0.8 (p < .05) (Figure 4 and Figure 5).

According to Boodhoo et al. (2009),\textsuperscript{[40]} probe geometry has to be adjusted to the hardness of the analyzed products. They found that a cylindrical probe with a needle load was better suited for analyzing firm fats, whereas a spherical probe delivered better results in analyses of soft fats. The cited authors concluded that the maximum penetration force is the most reliable measure of firmness and that this parameter is highly correlated with the results of penetration tests involving a cone probe.

**Textural parameters determined with the TA.XT. Plus texture analyzer and the P/S cylinder probe in the penetration test**

The mean values of the textural parameters determined with P/5CP in the penetration test are presented in Figure 6. According to the specification of the TA.XT. Plus texture analyzer, the area under the force-time curve represents sample softness (Figure 2). The smaller the area under the curve, the softer the sample. Jaworska et al. (2003)\textsuperscript{[31]} observed that this parameter can be a reliable indicator of a product’s consistency. In the current study, a highly significant positive correlation (r = 0.6285; \( p < .05 \)) was observed between the firmness and softness of butter samples measured with the TA.XT. Plus texture analyzer. The texture profile of butter, namely the structure and consistency of butter samples, is influenced by the crystallization behavior of milk fat and the size and quality of crystals formed during cream ripening.\textsuperscript{[4,8,9,15,16,57–60]}

Resistance to probe withdrawal or adhesiveness is also an important textural parameter. The mass of the sample that adheres to the probe after its withdrawal is responsible for the negative area under the force-time curve which denotes the adhesiveness of butter intended for spreading (Bobe et al. 2003).\textsuperscript{[42]} Adhesiveness was expressed as the product of the force necessary to withdraw the probe from the sample and withdrawal time [N-s]. Firmness and softness had no significant influence on the adhesiveness of the examined butter samples. Firmness and adhesiveness were bound by a weak negative correlation (r = -0.2084). In general, adhesiveness increases with a rise in firmness. According to other authors, the adhesive force that counteracts probe withdrawal is higher in samples with a cohesive structure.\textsuperscript{[42,61,62]} Chiavaro et al. (2007)\textsuperscript{[62]} noted that the presence of a crystalized milk fat fraction results in a high friction force. In their study, the adhesiveness of the analyzed emulsions increased with a rise in the content of milk fat.
CONCLUSION

The results of the analyses involving all instruments and probes were bound by high correlations in the range of $r = 0.7–0.9$ at $p < .05$. These observations suggest that all of the tested probes can be used to reliably measure the firmness of butter samples. The regression curves well fit the experimental data, and the highest agreement in the range of $R^2 = 0.7–0.8$ ($p < .05$) was observed between firmness values measured with WCP and P/5CP (TA.XT. Plus) (70.49%) and penetration values measured with AP4/2 CP and P/5CP (TA.XT. Plus) (79.96%). Measurements of textural attributes are more complex and laborious in shear tests than in penetration tests. The cone penetration test is undoubtedly the fastest and the simplest method of analyzing the textural properties of butter. Cone penetrometers are relatively cheap, and they generate repeatable results if sample temperature is carefully controlled. The results of this study indicate that the values of the textural properties of butter measured with different analysis systems can be compared. Penetration tests conducted with the use of a cone probe or a cylinder probe are rapid and easy to perform, and they can effectively replace shear tests.

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