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STORAGE STABILITY OF RAW MILK SUBJECTED TO VIBRATION

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Key words: raw milk, vibration, cold storage, chemical composition, physicochemical properties

The aim of the present study was to determine the effects of cold storage (temp. 4 and 8°C) on some physicochemical properties and technological suitability of raw milk subjected to vertical vibration at various frequencies (10, 30 and 60 Hz).

An analysis of the results showed that vibration increased milk acidity and negatively affected milk components. The process caused, among others, an increase in the levels of ionic calcium and free fatty acids, as well as protein degradation (an increase in the formalin number), which deteriorated the technological quality of milk (a decrease in thermal stability and ethanol stability, a shorter rennet coagulation time). The degree of these changes corresponded to an increase in vibrational frequency. Intensive lipolytic and proteolytic processes and enhanced acidity were observed during cold storage of raw milk subjected to vibration. This had a negative effect on the technological suitability of milk. The changes were less significant in the case of deep chilling (4°C).

INTRODUCTION

The quality attributes of milk are affected not only by genetic, physiological, health-related or dietary factors, but also by the sanitary conditions during milking and further handling of the raw material [Harding, 1999]. The standards concerning the production, storage and transportation of raw milk are specified in the EU Directive (Directive of the European Council 92/46/EEC of June 16, 1992) and the regulation by the Minister of Agriculture and Rural Development of May 18, 2005 (Dz. U. Nr 96, poz. 819).

Milk has a long way to go from the producer to the dairy plant, so the conditions during the transportation process are of great importance in terms of milk quality preservation. Even when both thermal and sanitary conditions are very good over transportation, this process is accompanied by certain unfavorable phenomena, like mechanical interactions, mostly shocks and vibrations, which have a negative influence on particular components and final quality of the raw material [Palich, 1993; Warmińska *et al.*, 2003a]. It is impossible, for organizational and economic reasons, to supply milk to the dairy plant after each milking. Thus, milk has to be stored at low temperatures on the farm, sometimes even for several dozen hours. Moreover, milk supplied to the dairy plant is not always processed immediately. Cold storage protects milk against acidifying bacteria, but not against psychrotrophic bacteria, which is highly undesirable since the latter produce large amounts of thermoresistant proteo- and lipolytic enzymes [Harding, 1999; Stepaniak & Rukke, 2003]. In addition, during long-term cold storage of raw milk low temperatures induce changes in almost all of its components, regardless of microbiological changes [White, 2003; Harding, 1999; Stepaniak & Rukke, 2003].

The aim of the present study was to determine the effects of vertical vibration at various frequencies on the storage stability of raw milk.

MATERIAL AND METHODS

The experimental materials comprised bulk milk collected from autumn to spring at the Research Station of the University of Warmia and Mazury in Olsztyn. Milk was poured into a plastic 25 L container, which was filled to capacity, and then transported to the laboratory. After a preliminary physicochemical evaluation (with Bactoscan 8000S, Fossomatic 5000 and Milkoscan 4000), milk was standardized to 3.5% fat, chilled to 8°C±1 and poured into 525 mL plastic bottles, which were filled in 80%, and then subjected to vertical vibration at various frequencies, *i.e.* 10, 30 and 60 Hz, for 60 min, at a constant acceleration of 1 g, at ambient temperature (8±1°C). The vibration was simulated using a vibration generator (VEB Schwingungstechnik und Akustik WIB), and the levels of vibrational frequency were differentiated with a RC decade frequency generator, type PW-9, coupled to a Leistungsverstärker power amplifier 3 Ω, 50 W, 3 Hz – 40 kHz, type LV 102 (VEB Metra Mess und Frequenztechnik). Vibration measurements were performed using a piezoelectric sensor, type KD 13/08 774, coupled to a Schwingungsmessgerät oscillatory vibration meter, type VM 6 (VEB Metra Mess Frequenztechnik). After vibration milk samples were stored at 4°C and 8°C, and analyses were made after 24, 48 and 72 h of incubation, following preservation (1 mL 2% NaN_3 solution per L milk)

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and stabilization (temp. 20±1°C) of the samples. Two control samples were used in the experiment. The first was a sample of standardized milk not subjected to vibration (K_1), and the other – a sample of standardized milk subjected to vibration without storage (K_2). The experiment was performed in five replications.

The effects of vibration on milk stability during storage were determined based on active acidity (WTW inoLab Level 1 pH-meter), the levels of ionic calcium (WTW inoLab pH/ION Level 2 ionometer) and free fatty acids (FFA) according to the method developed by Deeth and Fitz-Gerald [Deeth *et al.*, 1976], the formalin number by the Walker method [Budsławski & Drabent, 1972], heat stability (HS) as the time of milk coagulation at 140°C, ethanol stability (ES) – through titration of 10 mL milk with 96% ethyl alcohol [Kruk *et al.*, 1979], and the rennet coagulation time (RCT) as described by Alais & Jolles [1964].

RESULTS AND DISCUSSION

The milk samples used in the experiment were characterised by normal chemical composition, acidity typical of raw milk, and high microbiological and cytological quality, so they fulfilled the requirements specified in the regulation by the Minister of Agriculture and Rural Development of May 18, 2005 (Dz. U. Nr 96, poz. 819).

In the present study we determined the effects of transportation (simulation of vertical vibration at various frequencies, at constant acceleration and constant exposure) and cold storage conditions on the physicochemical properties of milk.

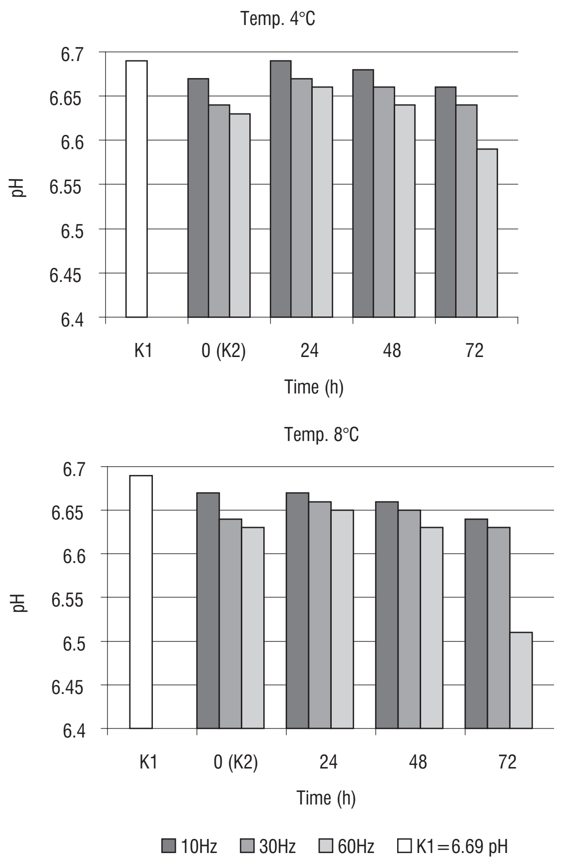

FIGURE 1. Effects of vibration and cold storage on the active acidity of milk (pH).

The level of acidity indicates freshness and determines technological suitability of milk. Vibration increased the active acidity of milk, and the greatest change in pH (a decrease by 0.06 unit) occurred at the highest vibrational frequency, *i.e.* 60 Hz (Figure 1). Changes in milk acidity caused by mechanical interactions can be related to changes in the proportions of salts and proteins. A special role is played by the level of soluble phosphates and citrates, and Ca^{2+} ions. Under the influence of vibration, part of micellar calcium phosphate passes to the soluble phase, thus increasing the concentration of Ca^{2+} ions and disturbing the structure of micelles, which considerably affects milk acidity [Palich, 1993; Warmińska *et al.*, 2003b]. More significant changes in active acidity were recorded during the cold storage of milk. Chilling for 24 h caused an increase in the pH of milk, proportional to the vibrational frequency. Greater changes took place in milk stored at a lower temperature (Figure 1). The process of alkalization was related to changes in the system of milk salts, *i.e.* greater solubility of calcium phosphates [White, 2003], and very good initial microbiological quality of the raw material (1×10^4 cfu/mL). During longer storage the pH of milk was gradually decreasing, and the highest increase in acidity (pH reduction by 0.12) was observed after 72 h of incubation at 8°C in milk samples subjected to vibration at a frequency of 60 Hz.

Vibration of milk samples contributed to increasing the level of ionic calcium, proportionally to the increase in frequency (Figure 2). Mechanical vibrations applied at the highest frequency, *i.e.* 60 Hz, caused an increase in the Ca^{2+} ion content of milk, by approx. 7% as compared with the control sample (K1). Greater changes in the level of ionic calcium were recorded over cold storage. After 72-h incu-

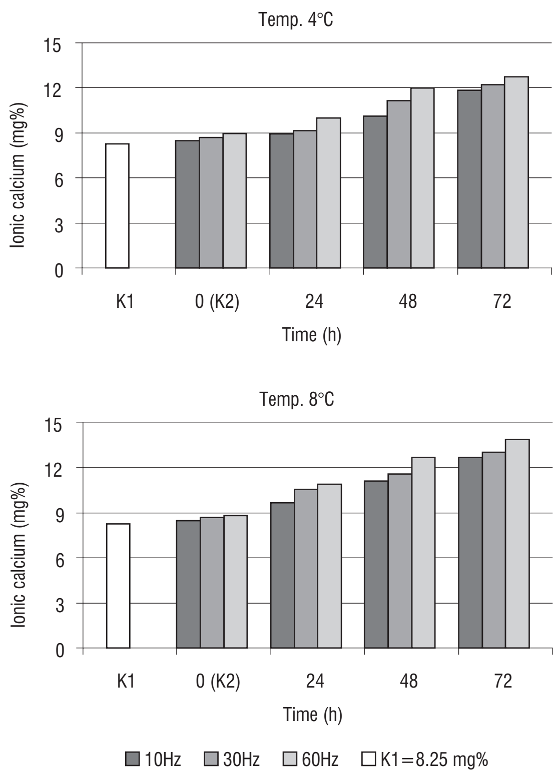

FIGURE 2. Effects of vibration and cold storage on the ionic calcium content of milk.

bation of milk samples subjected to vibration at 60 Hz the concentration of this calcium form increased by about 45 and 58% at 4 and 8°C, respectively. The changes in the ionic calcium content could be a consequence of the effect of the mechanical stimulus on the protein fraction of milk. Vibration propagating in the colloidal suspension induces vibrational resonance of colloid protein molecules. Ions of the diffusion layer of micelles have lower masses and a different coefficient of friction than micelles, so they undergo stronger period shifts, compared with the colloidal molecule, which may lead to both aggregation and disaggregation of proteins [Palich, 1993; Warmińska, 2003a]. On the other hand, changes in the level of Ca^{2+} ions during storage result from an increase in milk acidity, followed by changes in the structure of the casein complex. The decrease in milk pH makes colloidal forms of calcium turn into soluble compounds, which increases the level of ionic calcium [White, 2003].

Changes in the fat fraction of milk subjected to vibration and then stored are connected primarily with damage to the phospholipid-protein membranes of fat globules, conducive to the release of free fat, as well as to creaming and an increase in the concentration of free fatty acids [Warmińska & Kruk, 2001]. In milk subjected to vibration the level of free fatty acids showed a rising tendency as the vibrational frequency was increased (Figure 3). The maximum FFA content – 1.92 μEq/cm^3, *i.e.* an increase by approx. 54% as compared with the control sample (K_1), was recorded in milk samples subjected to vibration at 60 Hz. High dynamics of lipolysis was observed during cold storage of milk subjected to vibration. The highest FFA level was recorded after 72 h of cold incubation of milk samples subjected to vibration at 60 Hz. Compared with the control sample (K_1), the content of these acids increased by about 41 and 69% at 4°C and 8°C, respectively. Jurczak [1983] demonstrated that 5-min vibration at 1 g acceleration did not increase the FFA level in milk, but when the time of vibration exposure was extended to 10, 15 and 30 min, the concentration of FFA increased. Cold storage of milk causes changes in the fat fraction, related to solidification and fractioning of fat globules, and contributes to the proliferation of psychrotrophic bacteria, thus intensifying lipolytic processes [White, 2003].

The effects of vibration and cold storage of milk were also reflected by protein degradation (Figure 4). Proteolytic changes were determined on the basis of the so-called formalin number. Protein degradation (an increase in the formalin number) was directly proportional to the increasing vibrational frequency and prolonged time of cold storage. The most intensive proteolytic processes were observed during 72-h incubation of milk samples at 8°C. Then the formalin number of milk subjected to vibration at a frequency of 60 Hz increased by approx. 23%, in comparison with the control samples (K_1). These changes were a consequence of the destructive effect of vibration on protein compounds in milk [Palich, 1993; Warmińska *et al.*, 2003b] and of the proteolytic activity of the enzymatic system of psychrotrophic bacteria developing at low temperatures [Harding, 1999; White, 2003].

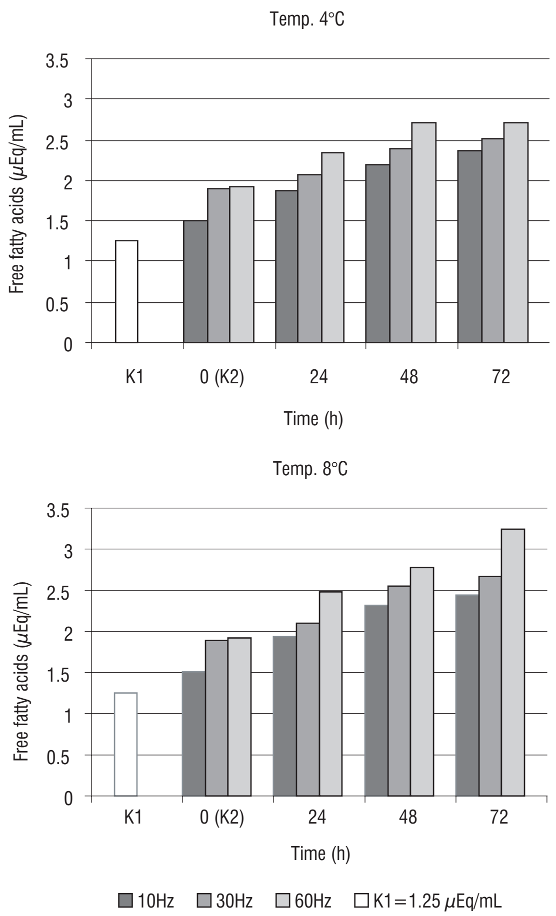

FIGURE 3. Effects of vibration and cold storage on the concentration of free fatty acids in milk.

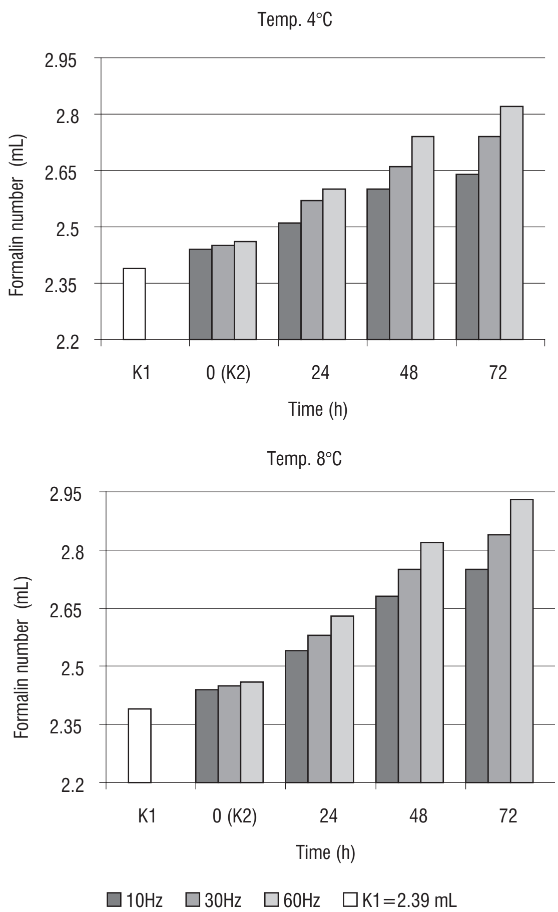

FIGURE 4. Effects of vibration and cold storage of milk on the formalin number.

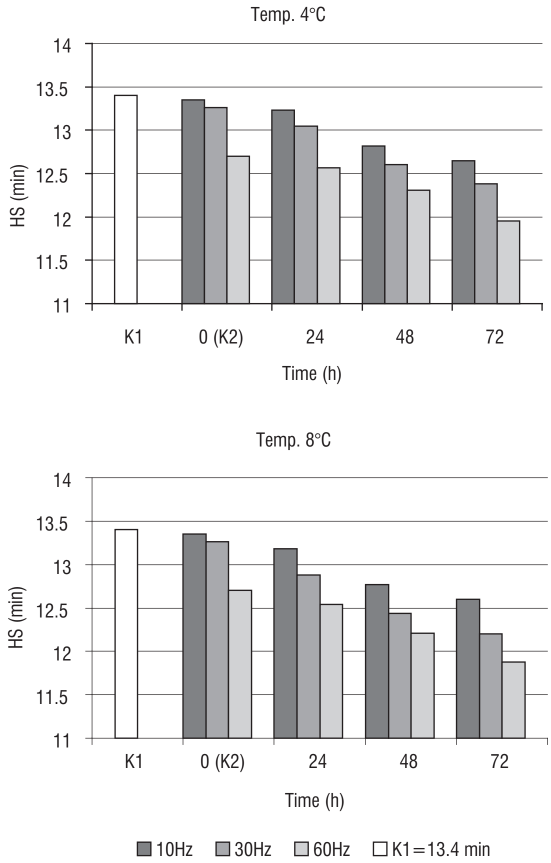

FIGURE 5. Effects of vibration and cold storage on the heat stability of milk.

The technological suitability of milk, including ethanol and heat stability or rennet coagulation, is determined, among others, by freshness, chemical composition and hygienic quality. The results of this experiment revealed the negative effects of vibrations and cold storage of milk on attributes of technological suitability. The influence of vibration on the stability of the colloidal system manifested itself by a reduction in both heat stability (Figure 5) and ethanol stability (Figure 6). This negative influence corresponded to the increase in vibrational frequency. Vibrations at a frequency of 60 Hz reduced the time of thermal coagulation of milk by approx. 5%, while ethanol stability decreased by about 10%, in comparison with the control sample (K_1). Greater changes in the heat and ethanol stability of milk were recorded during storage. Incubation at 8°C had a stronger negative effect. After 72 h of storage under these conditions, the heat and ethanol stability of milk samples subjected to vibration at 60 Hz decreased by approx. 12% and 32%, respectively. The destabilization of the colloidal system of milk was related to changes in milk components induced by vibration and chilling [Palich, 1993; Warmińska *et al.*, 2003a, b; White, 2003]. Key factors determining the colloidal stability of milk are the ionic environment of casein micelles (H^+, Na^+, Ca^{2+}, Mg^{2+}, citrate and phosphate ions), affected by the levels and proportions between soluble forms of milk salts, and the content and proportions of proteins (casein and its fractions, whey proteins, non-protein nitrogen compounds) [Singh, 1988; Czerniewicz *et al.*, 1999].

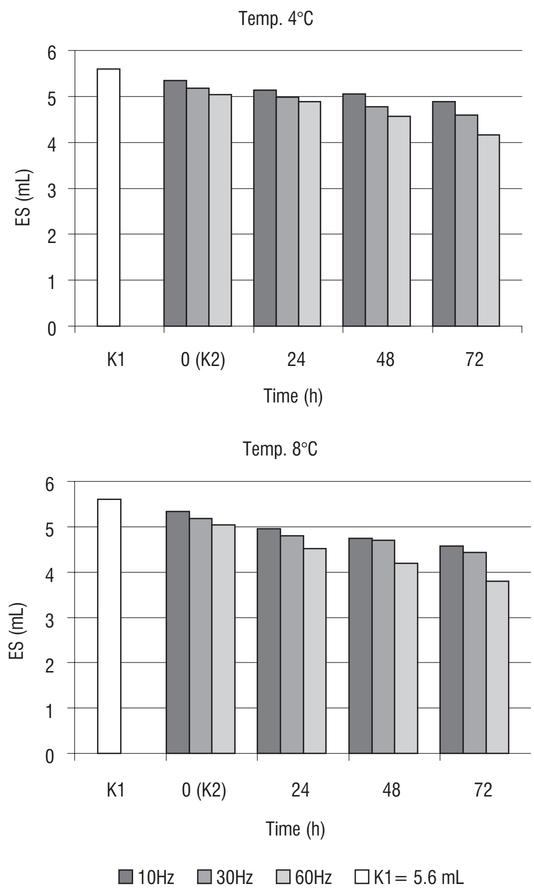

FIGURE 6. Effects of vibration and cold storage on the ethanol stability of milk.

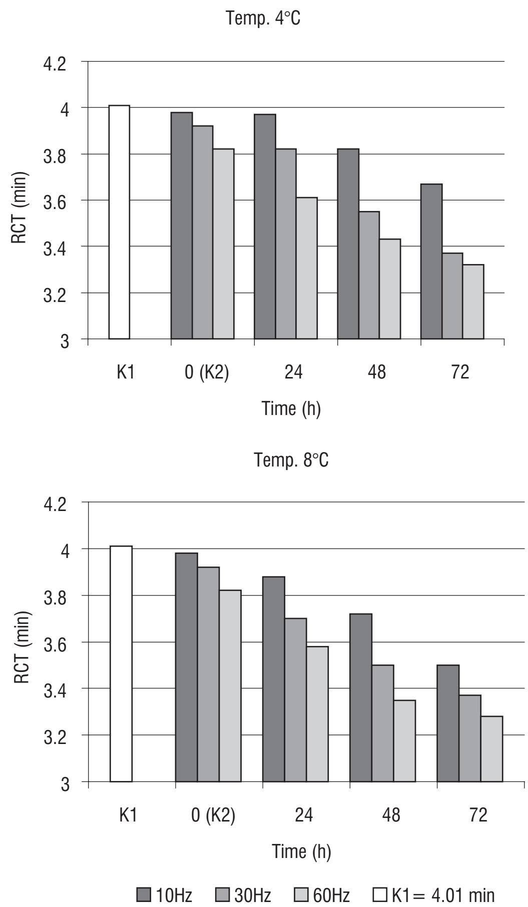

FIGURE 7. Effects of vibration and cold storage of milk on the rennet coagulation time.

As concerns the effect of vibration on the rennet coagulation time, it was found that RCT was gradually shortened along with an increase in vibrational frequency and prolonged time of cold storage (Figure 7). Vibration at the highest frequency (60 Hz) reduced the rennet coagulation time by about 5%, whereas 72-h cold storage of milk at 8°C made this tendency even more pronounced (RCT was shorter by about 18%). In the process of rennet coagulation of milk a key role is played by the concentration of calcium ions, the size of casein micelles and proportions of particular casein fractions, affected by milk pH. Changes in milk proteins under the influence of vibration, *i.e.* changes in casein hydration and size of micelles, contribute to the shortening of RCT [Palich, 1993]. On the other hand, cold storage of milk reduces the negative charge of micelles and decreases the degree of their hydration, thus shortening RCT as a result of dissociation of casein micelles, an increase in the levels of soluble casein (mainly at the initial stage of storage) and ionic calcium, and pH reduction [Stepaniak & Rukke, 2003; White, 2003].

CONCLUSIONS

1. Vibration negatively affected milk components, thus deteriorating its technological suitability. The degree of these changes corresponded to the increase in vibrational frequency.

2. Intensive lipolytic and proteolytic processes, as well enhanced acidity, were observed during cold storage of raw milk subjected to vibration. This had a negative effect on the technological quality of milk. The changes were less significant in the case of deep chilling (4°C).

3. Mechanical interactions and prolonged (72 h) cold storage of raw milk had a negative effect on its quality and technological suitability, making it useless especially for the production of durable dairy products.

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STABILNOŚĆ PRZECHOWALNICZA MLEKA SUROWEGO PODDANEGO WIBRACJI

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W pracy podjęto badania, których celem było określenie wpływu chłodniczego przechowywania (temp. 4 i 8°C) mleka surowego, uprzednio poddanego wibracji pionowej o zróżnicowanej częstotliwości drgań (10, 30 i 60 Hz), na wybrane cechy fizykochemiczne surowca oraz jego przydatność technologiczną.

Analiza wyników wykazała, że wibracja podwyższa kwasowość mleka (rys. 1) i niekorzystnie oddziałuje na jego składniki, powodując m.in. wzrost poziomu wapnia jonowego (rys. 2), zawartości WKT (rys. 3), degradację białek (wzrost miana formolowego – rys. 4), co skutkuje pogorszeniem jakości technologicznej surowca (obniżenie stabilności cieplnej – rys. 5 – i etanolowej – rys. 6, skrócenie czasu koagulacji podpuszczkowej – rys. 7). Stwierdzone zmiany analizowanych cech mleka pogłębiały się wraz ze wzrostem częstotliwości drgań. Podczas chłodniczego przechowywania mleka surowego, uprzednio poddanego wibracji, stwierdzono nasilenie procesów lipolitycznych (rys. 3) i proteolitycznych (rys. 4) oraz wzrost kwasowości (rys. 1), co wpłynęło negatywnie na jego przydatność technologiczną (rys. 5, 6 i 7), a mniejszy zakres tych przemian miał miejsce przy głębokim schłodzeniu surowca do temp. 4°C.