

EFFECT OF HOMOGENIZATION ON MILK CONDUCTANCE PROPERTIES*Joanna Katarzyna Banach¹, Ryszard Żywica¹, Katarzyna Kielczewska²**¹Chair of Commodity Industrial, Basic of Technique and Energy Management, ²Chair of Dairy Industry and Quality Management; University of Warmia and Mazury in Olsztyn, Olsztyn*

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The manuscript presents results of investigations aimed at determining changes in conductance properties of raw milk homogenized at pressures of 5, 10, 15 and 20 MPa.

Results obtained demonstrated that values of impedance were the highest and those of admittance – the lowest at the applied pressure of ca. 15 MPa, at which additionally the degree of fat globules disintegration appeared to approximate the maximum one (obtained at a pressure of 20 MPa). It was also concluded that the results achieved in the study constitute a basis for further research into the application of electric properties of milk in determination of the degree of fat globules dispersion in the homogenization process of raw milk.

INTRODUCTION

The production of high quality milk is one of the most difficult areas of animal production, since by its nature milk is highly susceptible to any physicochemical, microbiological and cytological changes [Ziajka, 1997]. In determining usability of raw milk for further processing a key role is ascribed to methods of assessing its quality attributes as well as to control and measuring equipment applied in those methods [Jurczak, 1999]. Technical advance, automation of production and requirements referring to milk prompt the need for modernization of quality control methods both for raw materials as well as for ready dairy products and for the efficiency of technological operations introduced at particular stages of the production process.

In the dairy industry, in order to prevent the phenomenon of creaming during long-term storage of liquid dairy products (drinking milk, sweet cream), use is made of homogenization. That treatment consists in the reduction and unification of sizes of fat globules in milk, which leads to their equal dispersion and, consequently, to increasing the stability of milk emulsion [Kielczewska & Kruk, 1997]. The efficiency of homogenization has so far been determined with a microscopic method [PN-A-86003/A.;1998; PN-79/A-86004:1979].

Recently, an increasing interest has been observed in instrumental methods of food quality assessment [Szabó, 1994] based, among other things, on measurements of its electric properties. The basic arguments speaking for the application of those methods are, above all: short time and accuracy of measurements performance, a lack of by-products detrimental to environment and an increasing number of adulterations.

What is more, automation of measurements enables shortening the time of their performance and increasing repeatability of results obtained.

The use of electric properties for elaboration of methods for quality assessment and control of such food products as milk poses a variety of difficulties linked, in particular, with the effect of such factors as: temperature, acidity, chemical composition or degree of fat globules disintegration. Though the effect of temperature and acidity of milk has been extensively described in literature [Żywica *et al.*, 2002; Mabrook & Petty, 2003a,b] data is lacking on the impact of fat globules size on its electric properties.

Taking the above into account, a study was undertaken that was aimed at determining the effect of fat globules disintegration on changes in conductance properties of raw milk subjected to the process of homogenization at pressures of 5, 10, 15 and 20 MPa.

MATERIAL AND METHODS

The experimental material was raw bulk milk (n=7) originating from the area of the Warmia and Mazury Province. The selection of milk samples was carried out based on: somatic cell count ($\leq 400 \times 10^3/\text{mL}$) determined with FOSSOMATIC type 5000; active acidity (pH=6.68) determined with an HI 8314 pH-meter; potential acidity (6.29°SH) against phenolphthalein; density (1.029 g/mL) with the aerometric method; and content of fat determined with the method of Gerber [PN-ISO-2446:2002].

Full fat milk was centrifuged at a temperature of $45 \pm 1^\circ\text{C}$ in an LWG 24E centrifuge (Spomasz, Gniezno, Poland), and the obtained skim milk and cream were cooled to a temperature

of $20 \pm 1^\circ\text{C}$ and used for the normalization of milk fat to be analysed. Afterwards, the milk was normalized to a fat content of 3.6% and subjected to homogenization at a temperature of $64 \pm 1^\circ\text{C}$ and pressures of 5, 10, 15 and 20 MPa in a U/G20 homogenizer (Rannie). A control sample was made of normalized milk not subjected to the homogenization process.

The analysed samples of milk, prepared according to the Polish Standard [PN-A-86059:1975] after prior preservation (1 mL of a 2% NaN_3 solution per 1 L of milk) and stabilization (temp. $20 \pm 1^\circ\text{C}$), were determined for the size of fat globules as Sauter diameter – d_{vs} [Juśkiewicz *et al.*, 1995] based on results of microscopic observations as well as for impedance (Z) and admittance (Y) values.

For measurements of impedance and admittance, milk (200 mL) was poured into glass containers (of own construction) with the size of $75 \times 55 \times 94$ mm, equipped with two plate electrodes made of acid-proof steel, mounted tightly to two opposite walls of containers (with smaller sizes). Next, the samples were fixed in a MEMMERT air washer to obtain a desirable temperature (20°C). Once the temperature has been settled, an HP 4263B measuring instrument was used at a constant voltage of 200 mV and frequency range of 100-100,000 Hz (100, 1000, 10,000 and 100,000 Hz) to measure the above-mentioned electric values ($n=3$). Analyses of electric properties (conductance) were carried out following an own-construction substitute diagram of raw materials and food products [Żywica *et al.*, 2005] presented in Figure 1. Experimental results obtained were discussed on the example of results of measurements performed at a frequency of 100 Hz.

A statistical analysis of the results achieved was conducted using a one-way analysis of variance ANOVA. The Duncan's test was applied to compare mean values. A coefficient of correlation between values of homogenization pressure and volume-surface diameter of fat globules of milk and the determined electric values was computed additionally using Excel software.

RESULTS AND DISCUSSION

The study demonstrated that values of milk impedance, at homogenization pressure increase from 5 to 15 MPa, increased from 97.75 to 105.76 Ω , *i.e.* by 7.6%. The greatest

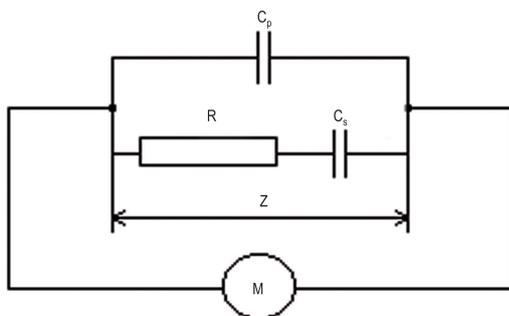


FIGURE 1. Scheme of a measuring system of electric properties of raw materials and food products: Z – impedance, R – resistance, C_s – equivalent serial capacitance, C_p – equivalent parallel capacitance, M – measuring device.

increase in impedance value, *i.e.* by 5.5%, was observed at pressure elevation from 10 to 15 MPa. Homogenization of milk at 20 MPa evoked a decrease of impedance to 96.26 Ω , *i.e.* to a value lower by *ca.* 1% than that of non-homogenized milk (97.37 Ω), (Figure 2).

Values of admittance of milk homogenized in a pressure range from 5 to 15 MPa decreased from 10.28 to 9.49 mS, *i.e.* by 8%, still those changes were observed to intensify along with pressure increase. The lowest value (9.49 mS) of admittance was recorded at a pressure of 15 MPa. An increase of milk homogenization pressure to 20 MPa resulted in an increase of admittance to 10.42 mS. That value was higher by *ca.* 1% than admittance value of milk not subjected to the homogenization process (10.30 mS), (Figure 3).

Results of analyses of the effect of volume-surface diameter of fat globules on changes in electric properties of raw milk indicated that, as a result of homogenization at a pressure of 5 MPa, diameter of fat globules decreased to the largest extent and the value of impedance was, practically, unchanged. The highest value of milk impedance (105.67 Ω) was noted at fat globules diameter reaching 1.12 μm , obtained upon homogenization pressure of 15 MPa. At a further (inconsiderable) decrease of fat globules diameter, upon pressure of 20 MPa, a rapid decline occurred in the value of impedance to 96.26 Ω (Figure 4).

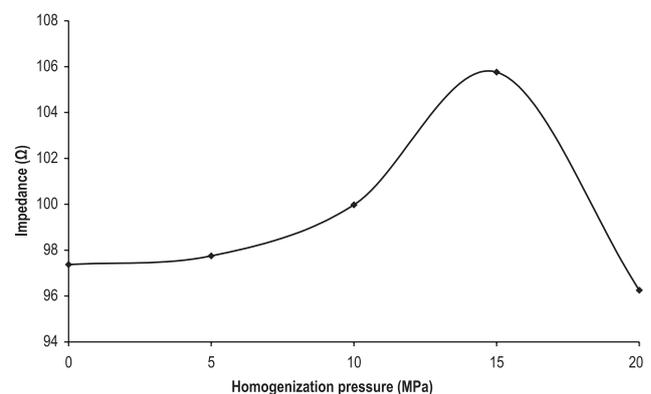


FIGURE 2. Changes in milk impedance depending on homogenization pressure applied.

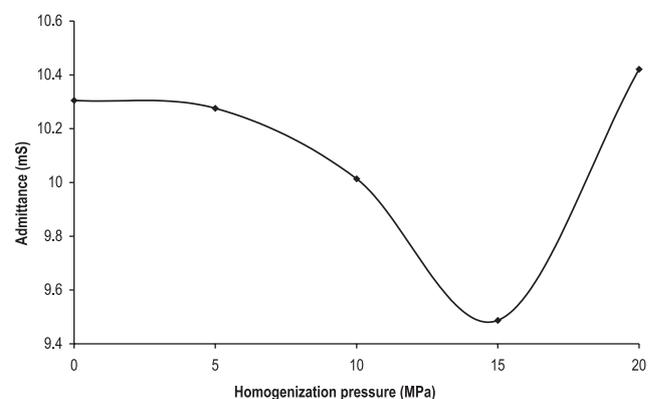


FIGURE 3. Changes in milk admittance depending on homogenization pressure applied.

During the initial, most distinct decrease of fat globules diameter (by *ca.* 60%), at a pressure of 5 MPa, the value of admittance, likewise that of impedance, remained unchanged. An increase of milk homogenization pressure to 10 MPa evoked subsequent disintegration of fat globules (by 17.6%), as a result of which conductance of the system (admittance) began to decrease till the moment when fat globules reached a diameter of $1.12 \mu\text{m}$ (homogenization pressure of 15 MPa). The value of admittance accounted then for 9.49 mS. A further decline in volume-surface diameter of fat globules along with pressure increase to 20 MPa resulted in a rapid increase of admittance. Its highest value (10.42 mS) was recorded at the lowest achieved volume-surface diameter of the fat globules reaching $1.07 \mu\text{m}$ (Figure 5).

Calculation of a correlation coefficient carried out in the study did not demonstrate any significant correlation between values of homogenization pressure and volume-surface diameter of fat globules and the examined electric values (Table 1).

Investigations carried out so far have indicated that milk is characterised by good electric conductance due to the presence of mineral salts dissociated in aqueous phase. Conductance is also determined by fat contained in milk, due to its poor specific electric conduction [Mabrook & Petty, 2003b]. In turn, homogenization affects a change in the primary structure of milk components by modifying emulsion and colloidal

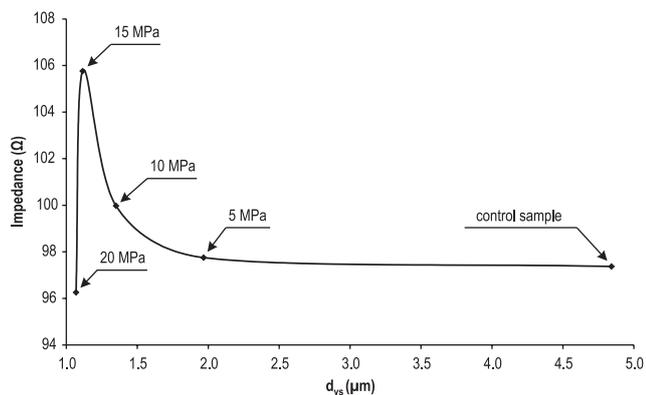


FIGURE 4. Changes in raw milk impedance depending on volume-surface diameter of fat globules (d_{vs})

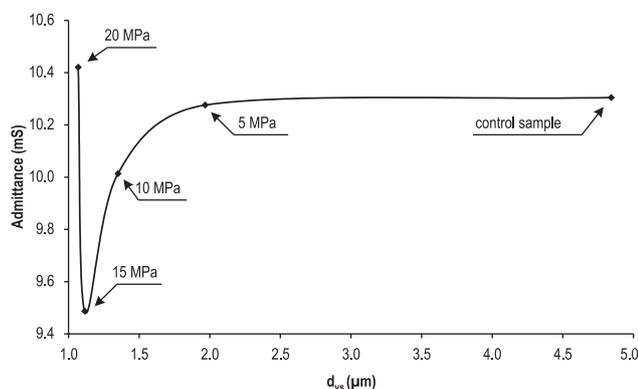


FIGURE 5. Changes in milk admittance depending on volume-surface diameter of fat globules (d_{vs})

TABLE 1. Coefficient of correlation between the examined properties of homogenized milk.

Electric values	Values of determined coefficients of simple correlation at a significance level of $\alpha \leq 0.05$	
	Pressure (MPa)	d_{vs} (μm)
Impedance (Ω)	0.241	-0.364
Admittance (mS)	-0.235	0.368

phases [McCrae *et al.*, 1994], thus contributing to changes in electric properties of full fat milk.

Results obtained in the presented study demonstrated that homogenization affected a decrease and unification of sizes of fat globules, which were observed to intensify along with an increasing pressure. It has been indicated by results collected in analyses of the volume-surface diameter of fat globules, expressed as d_{vs} . The d_{vs} value of fat globules, reflecting heterogeneity of their sizes, was found to decrease along with an increase of pressure. The most rapid decline in the average value of fat globules diameter occurred at pressure increase to *ca.* 15 MPa, yet a further increase of pressure to 20 MPa contributed to an increasing degree of fat dispersion only to a negligible extent [Marcinowska, 2006]. According to literature data [Kielczewska & Kruk, 1997], it may lead to flocculation and coalescence of fat globules and reduced stability of dairy product emulsions.

An increased degree of fat globules dispersion, *i.e.* an increase in their number and reduction of their sizes, has contributed to increased impedance of milk subjected to pressure treatment at 15 MPa. Different changes of that value were observed upon treatment at pressure above 15 MPa. Causes of that should be sought in the modification of the colloidal phase of milk, *i.e.* imbalance between components of the soluble and colloidal phases of milk. For homogenization, apart from increasing the degree of fat dispersion, triggers changes in the state of milk proteins which in the case of full fat milk are linked, most of all, with adsorption of milk plasma proteins, casein in particular, on the interfacial surface: plasma – milk fat [Dalglish & Robson, 1985; Michalski *et al.*, 2002].

Homogenization is known to influence disintegration of micelles, however it has not been elucidated so far whether it results either from a mechanical action that during homogenization evokes disruption of casein micelles into sub-units that are readily adsorbed on fat surface, or from the action of interfacial forces during casein adsorption on an increasing interfacial surface: fat – milk plasma [Garcia-Risco *et al.*, 2002]. A consequence of an increased degree of casein micelles dispersion and disintegration of micelles into subunits and their adsorption on milk fat surface upon homogenization is loss of balance of mineral salts linked with dissociation of colloidal calcium phosphate and an increased in the contents of calcium and phosphorus in milk serum (transformation of colloidal calcium phosphate into a soluble form). This has been confirmed in a study by Kielczewska *et al.* [2006] who demonstrated an increase in solubility of calcium and phosphorus salts as well as a decrease in their level in the colloidal form as a result of high-pressure homogenization of milk with 4% fat content. The content of mineral salts in milk and balance between their forms occurring in the colloidal and soluble form is of great significance in modeling milk

admittance and impedance. Mabrook & Petty [2003b] reveal that the disruption of phosphate-calcium bonds during casein micelles transition into sub-micelles, accompanied by an increased level of soluble salts, mainly of calcium, contributes to increasing conductance.

Taking into account the above literature data it may be assumed that the increased content of mineral salts present in molecular dispersion occurred as a result of homogenization run at a pressure above 15 MPa and was manifested by a considerable decrease of milk impedance. The effect of homogenization on electric properties of milk is linked, therefore, not only with changes in the degree of dispersion and size of fat globules, but also with the modification of the colloidal phase of milk, *i.e.* with imbalance between soluble and colloidal forms of calcium and phosphorus salts.

CONCLUSIONS

1. Negligible changes of impedance and admittance in homogenization pressure range of 5-10 MPa indicate that the degree of fat globules disintegration, corresponding to the volume-surface diameter ranging from 5 to 1.5 μm , had no effect on conductance properties of milk, which is likely to be of significance at applying those properties in analyses of the chemical composition and quality of raw milk.

2. Fluctuations in fat globule sizes or changes in the structure of casein, that control solubility and balance of mineral salts, impair the application of impedance and admittance measurements in the determination of the size of fat globules and degree of milk homogenization. Simultaneously they prompt to undertake more extensive research on the electric properties of milk.

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WPLYW HOMOGENIZACJI NA WŁAŚCIWOŚCI PRZEWODNOŚCIOWE MLEKA

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W pracy przedstawiono wyniki badań, których celem było określenie zmian właściwości przewodnościowych mleka surowego w procesie homogenizacji w zależności od stosowanego ciśnienia.

Materiał doświadczalny stanowiło pełne mleko zbiorcze ($n=7$) normalizowane do zawartości tłuszczu 3,6%, podgrzewane do temperatury $64 \pm 1^\circ\text{C}$ i poddawane homogenizacji przy ciśnieniu 5, 10, 15 oraz 20 MPa. Następnie mleko konserwowano (2% roztwór NaN_3 – 1 mL/1 L mleka) i schładzano do temperatury 20°C . Po schłodzeniu próbkach mleka, na podstawie wyników obserwacji mikroskopowych, określano wielkość kuleczek tłuszczowych oraz wykonywano pomiary impedancji i admitancji, miernikiem typu HP 4263B, przy napięciu 200 mV i częstotliwości od 100 Hz do 100 kHz.

Przeprowadzone badania wykazały, że największy wzrost wartości impedancji tj. o 5,5% zaobserwowano przy wzroście ciśnienia homogenizacji od 10 do 15 MPa. Zwiększenie ciśnienia do 20 MPa spowodowało zmniejszenie jej wartości do $96,26 \Omega$ tj. o ok. 1% mniejszej od wartości impedancji mleka nie poddanego procesowi homogenizacji ($97,37 \Omega$). Admitancja mleka, najniższą wartość ($10,42 \text{ mS}$) osiągnęła przy ciśnieniu 15 MPa. Przy ciśnieniu 20 MPa wartość admitancji wynosiła $10,42 \text{ mS}$ i była większa o ok. 1% w porównaniu do wartości admitancji mleka nie poddanego homogenizacji ($10,30 \text{ mS}$). Wyniki badań wykazały ponadto, że stopień rozdrobnienia kuleczek tłuszczowych w zakresie ciśnienia 5–10 MPa oraz przy ciśnieniu 20 MPa nie ma wpływu na właściwości przewodnościowe mleka surowego. Natomiast wzrost stopnia dyspersji kuleczek tłuszczowych mleka poddanego działaniu ciśnienia 15 MPa spowodował gwałtowny wzrost wartości jego impedancji oraz gwałtowne zmniejszenie wartości admitancji.

Na podstawie uzyskanych wyników stwierdzono, że istnieje podstawa prowadzenia dalszych badań nad wykorzystaniem właściwości elektrycznych mleka do oceny i kontroli stopnia dyspersji kuleczek tłuszczowych w procesie homogenizacji mleka surowego.