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Potassium-based emulsifying salts in processed cheese: A rheological, textural, tribological, and thermal approach

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ABSTRACT

The aim of the study was to investigate the effect of potassium-based emulsifying salts (ES; 2% wt/wt concentration) with different phosphate chain lengths (dipotassium hydrogenphosphate [K₂HPO₄; DKP], tetrapotassium diphosphate [K₄P₂O₇; KTPP], pentapotassium triphosphate [K₅P₃O₁₀; TKPP]) on the physicochemical, viscoelastic, textural, tribological, thermal, and sensory properties of processed cheese (PC; 40% wt/wt DM, 50% wt/wt fat in DM) during a 60d storage period (6°C \pm 2°C). On the whole, the hardness of all PC samples increased with the increasing chain length of ES (DKP < TKPP < KTPP) and the prolonging storage period. Moreover, the hardness results were in accordance with those of the rheological analysis. All PC samples exhibited a more elastic character (G' > G''; tan $\delta < 1$). The type of potassium-based ES affected the binding of water into the structure of the PC. Furthermore, the study confirmed that the manufactured PC received optimal sensory scores, without any excessive bitterness. It could be concluded that the type of applied ES and storage length affected the functional properties of PC. Finally, the information provided in this study could serve as a tool for the dairy industry to help appropriately select potassium-based ES for PC manufacture with desired properties.

Key words: processed cheese, potassium-based emulsifying salts, rheological properties, textural properties, thermal properties

INTRODUCTION

From a physicochemical point of view, the term "processed cheese" (PC) defines a complex multicomponent dairy system, also described as a stable oil in water emul-

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sion (Chen and Liu, 2012; Salek et al., 2017). Generally, PC refers to a dairy product with a relatively high-water content (50%–70% wt/wt) containing one or more types of natural cheeses (at varying degrees of maturity), as well as other ingredients of dairy (butter, cream, whey, whey powder, caseinate) and nondairy (emulsifying salts [ES], hydrocolloids, flavorings, colorings, preservatives) origin. Heating natural cheese (and other ingredients) under constant shear and partial vacuum in the presence of ES leads to the development of a final product with homogeneous and smooth consistency and extended shelf life (Johnson et al., 2009; Salek et al., 2016; Pluta-Kubica et al., 2021).

In addition, ES are key-ingredients in PC manufacturing. The most commonly applied ES for PC manufacture include especially sodium salts of phosphates, polyphosphates, citrates and, combinations of thereof (Salek et al., 2016; Sołowiej et al., 2020). In general, ES are salts of a monovalent cation (Na⁺ or K⁺) and a polyvalent anion (PO₄³⁻) enhancing the emulsifying and stabilizing properties of milk proteins (caseins). In the system of natural cheese, the caseins are bonded by calcium bridges in a 3-dimensional network. The latter crosslinking makes impossible for the caseins to imply their natural emulsifying properties, which are essential for emulsifying the fat and binding the water to ensure a smooth and homogeneous PC consistency (Guinee et al., 2004; Kapoor and Metzger, 2008; Chavhan et al., 2015). Furthermore, the primary function of ES during the manufacturing of PC (under the simultaneous action of heat and shear) is the exchange of sodium/potassium ions for calcium ions (in the natural cheese matrix). In particular, the latter phenomenon leads to the conversion of the insoluble calcium paracaseinate (natural cheese) to the more soluble sodium or potassium para-caseinate, whose molecules readily disperse in the protein system of the PC and coat the surface of the dispersed free fat released during heating of the natural cheese. Thus, the dispersed casein significantly increases fat emulsifica-

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The list of standard abbreviations for JDS is available at adsa.org/jds-abbreviations-24. Nonstandard abbreviations are available in the Notes.

tion and water binding in the cheese matrix. As a result, ES can be considered as a key-ingredient in PC manufacturing, resulting in a series of coordinated physicochemical changes in the cheese matrix: calcium sequestration, casein and solubilization peptization, protein hydration and swelling, water binding and stabilization. However, not all ES have the same ion-exchange ability. Generally, the ability to support ion exchange occurs in the following order (when comparing sodium-based ES): citrates \approx monophosphates < diphosphates < triphosphates < short-chain polyphosphates (<10 phosphorus atoms in a molecule) < long-chain polyphosphates (>10 phosphorus atoms in a molecule; Nagyová et al., 2014; Salek et al., 2015; 2016).

The type and concentration of the ES used also affects the functional (viscoelastic, textural, tribological) and, sensory properties of the resultant PC, which are used to characterize PC consistency (Kapoor and Metzger, 2008; Lucey et al., 2011; Sudhakar et al., 2020). In particular, viscoelastic measurements are used to characterize the deformation (mostly small) behavior of PC when subjected to stress. In addition, PC contain moisture and solids (proteins, fats, carbohydrates) and exhibit the properties of both elastic and viscous materials, therefore, they can be classified as viscoelastic materials (Sutheerawattananonda and Bastian, 1998). Moreover, textural measurements provide information about the response of PC to large deformations, commonly during 2 successive penetration events simulating process of mastication. The main textural parameters used for PC include hardness, cohesiveness, relative adhesiveness, chewiness, and gumminess (Fox et al., 2017). Additionally, tribological measurements provide insight about PC when simulating oral processing in the mouth (between the surfaces of the tongue and palate), including an assessment of friction and lubrication. In general, the basic principle of tribology (also known as surface rheology) is to simulate the surface roughness of the human tongue based on the friction and lubrication of 2 surfaces as they move together (Nguyen et al., 2016a; Sudhakar et al., 2020).

A general trend in modern nutrition is the sodium content reduction in the diet, because high sodium intake and insufficient potassium intake can contribute to the possible development of cardiovascular diseases. PC have a relatively high in sodium content (usually >1% wt/wt) in the form of NaCl and sodium-based ES (which are the most commonly applied). Recently, the possibility of replacing commonly used sodium-based ES mixtures with potassium-based ES has been investigated. However, up to now only a few studies have dealt with the above-mentioned issue. Hence, the appropriate choice of potassium-based ES type and concentration is crucial (Agarwal et al., 2011; Hoffmann et al., 2012; Chavhan et al., 2015). On the whole, the available scientific literature is missing a study providing a complex and systematic characterization of PC made with potassium-based ES, with the use of rheological, textural, tribological, and thermal techniques.

Thus, the objective of the present study was to evaluate the effect of potassium-based ES (applied as sole ingredients in a concentration of 2% wt/wt) with different phosphate chain lengths (dipotassium hydrogenphosphate [K₂HPO₄; **DKP**], tetrapotassium diphosphate [K₄P₂O₇; **KTPP**], pentapotassium triphosphate [K₅P₃O₁₀; **TKPP**]) on the physicochemical, viscoelastic, textural, tribological, thermal and sensory properties of PC during a 60-d storage period (6°C \pm 2°C).

MATERIALS AND METHODS

Ingredients for Manufactured-PC Samples

The following ingredients were used to manufacture the model PC samples: (1) Edam cheese blocks (52% wt/ wt DM content, 30% wt/wt fat in DM [**FDM**], a Dutchtype semihard cheese; 7-wk maturity); (2) unsalted butter (83% wt/wt fat content, 85% wt/wt DM content), (3) water and (4) ES (applied as sole ingredients). Three different potassium-based ES were used: dipotassium hydrogenphosphate (K₂HPO₄; DKP); tetrapotassium diphosphate (K₄P₂O₇; KTPP); pentapotassium triphosphate (K₅P₃O₁₀; TKPP). All potassium-based ES were used in a concentration of 2% wt/wt. The composition of the used ingredients is shown in Table 1.

Model of the Manufactured-PC Samples

The model PC samples were designed to have target DM and FDM contents of 40% and 50% wt/wt, respectively. Moreover, the samples were prepared with a Niromix blender cooker equipment (5-L capacity; Nirosta s.r.o., Chlumec nad Cidlinou, Czech Republic) with indirect heating. First, natural cheese and butter were cut into pieces (20 mm \times 20 mm \times 20 mm). Thereafter, natural cheese was placed in the apparatus for disintegration (60 s; 3,000 rpm). Then the rest of the ingredients were added (butter, ES, and water) into the blender cooker vessel. The target melting temperature was set at $90^{\circ}C \pm 1^{\circ}C$ with a holding time of 3 min. The total processing time was ~ 12 min. The hot melt was poured into laminated aluminum containers (conical shape; inner dimensions of 26.8 mm in height, 81.1 mm in diameter at the top, and 68.9 mm in diameter at the bottom) and sealed with aluminum lids with the NovaSeal equipment (Nirosta s.r.o., Chlumec nad Cidlinou, Czech Republic). The weight of the sample in one container was $\sim 93 \pm 3$ g (from each manufacturing batch ~28 containers were obtained). The

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Ingredient	DM content (% wt/wt)	Fat content (% wt/wt)	Protein content (% wt/wt)	Processe	d cheese (rela emulsifying	ative %), salt
Edam cheese ¹	52.08	15.32	31.17	50.81	50.81	50.81
Butter	84.56	83.14	0.00	15.25	15.25	15.25
Water	0.00	0.00	0.00	31.94	31.94	31.94
Emulsifying salts ²						
K ₂ HPO ₄	>95	0.00	0.00	2.00	0.00	0.00
$K_4 P_2 O_7$	>95	0.00	0.00	0.00	2.00	0.00
$K_{5}P_{3}O_{10}$	>95	0.00	0.00	0.00	0.00	2.00

 Table 1. Formulation of the processed cheese samples manufactured with different potassium-based emulsifying salts and with target values: 40% wt/wt DM content, 50% wt/wt fat in DM content, and 14% wt/wt protein content

¹Dutch-type semihard cheese, 7-wk maturity.

²Composition information was obtained from the manufacturer of emulsifying salts.

PC samples were left to cool (at $25^{\circ}C \pm 2^{\circ}C$) and then were stored ($6^{\circ}C \pm 2^{\circ}C$) until analyses were realized. Each PC sample was manufactured in triplicate (3 types of potassium-based ES × 3 repetitions = 9 lots). The PC products were analyzed on d 1, 14, 30, and 60 of storage ($6^{\circ}C \pm 2^{\circ}C$; d 0 was the day of the manufacture). Exception was the proximate analysis, water activity determination, atomic emission spectrometry and instrumental color measurement (performed on d 1) and tribological analysis (performed on d 30).

Proximate Analysis, pH, and Water Activity Determination of PC

Dry matter, fat, and protein contents were determined according to ISO 5534:2004 (ISO, 2004), ISO 23319:2022 (ISO, 2022), and ISO 8968–1:2014 (ISO, 2014), respectively after 1 d of storage. The analyses were performed at least 6 times (n = 6) for each tested PC sample.

The pH of the samples was determined using a Foodcare pH meter (HI-99161, Hanna Instruments Inc., Woonsocket, RI) with a combined glass tip electrode at $20 \pm 1^{\circ}$ C. The pH values were determined by inserting a glass tip electrode of pH meter directly into the PC samples (n = 6). Before the analysis the tested PC samples were tempered for 4 h at laboratory temperature and the final temperature were controlled by the thermometer (HI935004, Hanna Instruments Inc., Woonsocket, RI).

Water activity (\mathbf{a}_w) was determined using a Meter AquaLab 4TE (AquaLab, Decagon). Before each measurement, the instrument was calibrated with a suitable solution ($\mathbf{a}_w = 0.92$ NaCl 2.33 molar in H₂O [Qi Analytical s.r.o., Prague, Czech Republic]). The analyses were performed at least 6 times (n = 6).

Dynamic Oscillatory Rheology of PC

The determination of the PC samples viscoelastic properties was performed using a dynamic oscillatory shear rheometer (Thermo Scientific RheoStress 1, Haake, Bremen, Germany) with a plate-to-plate geometry (35 mm in diameter) at 20 ± 0.1 °C and a gap of 1 mm was used. The amplitude of shear stress (20.0 Pa) was selected in the linear region of viscoelasticity. All tested PC samples were measured in the control stress mode at a frequency range of 0.1 to 100.00 Hz. Moreover, to avoid sample dehydration a thin layer of silicon oil (EKOLUBE, s.r.o., Brno, Czech Republic) was used to cover the exposed edges of the plate-to-plate geometry. The monitored parameters included the storage (G') and loss (G") moduli (determined as a function of frequency) and were used for complex moduli (G*) calculation according to the Equation [1]:

$$G^* = \sqrt{\left(G'\right)^2 + \left(G''\right)^2}.$$
 [1]

In addition, the loss tangent ($tan \ \delta = G''/G'$) was also calculated. The values of G* and $tan \ \delta$ were presented for the reference frequency of 1 Hz. Analyses were performed at least 6 times for each tested PC sample (n = 6).

TPA of PC

The texture profile analysis (**TPA**) of the PC samples was performed using a TA.XTplus texture analyzer (Stable Micro Systems Ltd., Godalming, UK). Moreover, the tests were carried out at $6 \pm 2^{\circ}$ C (measurements were performed immediately after removing the samples from a refrigerator where they were stored). Texture profile analysis was implemented by 2 successive (bites) of compression-decompression cycles with a 20 mm diameter stainless steel cylindrical probe (cylindrical shape; 15 mm diameter and 20 mm height; were made from molds and the upper surface of the tested samples was cut carefully to eliminate surface irregularities). The depth of probe was set at 10 mm at a speed rate of 2 mm s⁻¹; the trigger force corresponded to 5 g and the time between the 2 cycles was set at 5 s). The data for force as

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Table 2. Results of the analysis of the processed cheese samples manufactured with different potassium-based
emulsifying salts after 1 d of storage ($6 \pm 1^{\circ}$ C; results were expressed as mean \pm SD)

	Processed cheese				
Parameter	DKP	ТКРР	KTPP		
DM content (% wt/wt)	$40.18\pm0.45^{\rm a}$	$40.52\pm0.51^{\mathrm{a}}$	$40.32\pm0.47^{\rm a}$		
Fat content (% wt/wt)	$20.09\pm0.05^{\rm a}$	$19.97 \pm 0.08^{ m b}$	$20.05 \pm 0.03^{a,b}$		
Fat in DM content ¹ (% wt/wt)	50.02	49.98	50.08		
Lightness (L*)	$92.12\pm0.57^{\rm a}$	$92.07 \pm 0.03^{\rm a}$	$92.01 \pm 0.14^{\rm a}$		
Chromaticity on a green to red axis (a*)	$0.33\pm0.05^{\rm a}$	$0.28\pm0.02^{\rm a,b}$	$0.18\pm0.03^{\rm b}$		
Chromaticity on a blue to yellow axis (b*)	$14.46\pm0.68^{\rm a}$	13.22 ± 0.13^{b}	$13.28 \pm 0.12^{b,c}$		
Na content (mg kg ^{-1})	$4,505.1 \pm 351.9^{a}$	$9,136.3 \pm 64.7^{b}$	$8,970.3 \pm 250.9^{ m b,c}$		
K content (mg kg ^{-1})	$20,618.6 \pm 554.8^{a}$	$21,759.0 \pm 157.7^{b}$	$19,958.6 \pm 539.6^{\circ}$		
Mg content (mg kg ^{-1})	$482.3\pm21.9^{\rm a}$	409.3 ± 22.5^{b}	$422.6 \pm 18.9^{b,c}$		
Ca content (mg kg ⁻¹)	$6{,}966.3 \pm 337.6^{a}$	$7,\!178.0\pm675.8^{\rm a}$	$7,098.6 \pm 293.2^{a}$		

^{a-c}Means within a row with different superscripts differ (P < 0.05).

¹The fat in DM content values were calculated.

a function of time were obtained for the 2 compression– decompression cycles, and using the "texture profile" function of the Texture Exponent Lite software (Stable Micro Systems Ltd., Godalming, UK), and values for the following parameters were obtained: hardness, cohesiveness, and relative adhesiveness. All tested samples were measured at least 6 times (n = 6).

Tribological Analysis of PC

Tribological measurements of PC were performed using a TBR3 Pin-on-Disk Tribometer (Anton Paar Czech Republic s.r.o., Prague, Czech Republic) on a rough plastic surface (3M Transpore surgical tape 1527-0; 3M Health Care), simulating the roughness of the surface of the human tongue. According to Nguyen et al. (2016b), the above-mentioned tape has similar wetting and surface roughness to the human tongue. The tape was cut into a rectangular shape (13 mm \times 60 mm), positioned and pressed firmly on the top of the bottom plate geometry on which the PC sample (of ~ 2 g) was applied and was sufficient to cover the tape with a layer of ~2-mm thickness. After each measurement, the tape was replaced and the surface was cleaned with deionized water and dried with laboratory wipes. The tested PC samples were left to equilibrate at room temperature (25°C) for 4 h before the tribological measurements were performed under the appropriate simulation conditions of an oral environment at 37°C with a selected axial force of 1 N, a travel distance of 0.94 m over 50 cycles and 200 rpm. The frictional (lubrication) properties of PC samples were determined as coefficient of friction (CoF; the ratio of friction stress and normal stress) as $\mu 1/4$ dF/dN, where F 1/4torque (Nm) and N 1/4 1N. The friction curves were obtained by plotting CoF against sliding speed. Minimum CoF (CoF_{min}) and initial CoF (CoF_i) were obtained from the graph. Samples were pre-sheared at

 0.01 s^{-1} for 1 min and then equilibrated for an additional 1 min before each measurement. All measurements were performed at least 6 times (n = 6).

Atomic Emission Spectrometry of PC

An atomic absorption spectrometer (contrAA 800D; Analytik Jena, Jena, Germany) was used to determine the amount concentrations of potassium, sodium, calcium, and magnesium in the tested model PC samples. First, the PC samples (0.25 g of sample) were mineralized by adding 6 mL of concentrated nitric acid (65% vol/vol) and 2 mL of hydrogen peroxide (30% vol/vol). Mineralization process was then performed using an Ethos SEL Microwave Labstation (Milestone, Sorisole, Italy) at 200°C for 30 min. The amount of potassium was determined at wavelengths of 588 and 766 nm using flame emission spectrometry with an air-acetylene flame composition. Additionally, calcium and magnesium were determined by absorption at wavelengths of 422.7 nm and 285.2 nm. All measurements were performed at least 3 times (n = 3) after 1 d of storage. Furthermore, the obtained data were processed using the Aspect CS software (ver. 2.2.1.0; Analytik Jena, Jena, Germany), with the final result being one final value for each sample (batch of product; Kameník et al., 2017; Macharáčková et al., 2021).

Instrumental Color Measurement of PC

Instrumental color evaluation of the PC samples was performed by using the HunterLab UltraScan VIS Pro spectrophotometer (Hunter Associates Laboratory, Inc., Reston, VA) at $25 \pm 1^{\circ}$ C with a with a glass cuvette. The CIELAB color scale (L*a*b*) with illuminant D65 (average daylight) and observer angle of 10° was used. Before the measurements, it was necessary to calibrate the instrument in the reflectance mode, excluding specular



Figure 1. The development of processed cheese pH (A), hardness (N; B), complex modulus (G*; Pa; for the reference frequency of 1 Hz; C), and tangent delta (for the reference frequency of 1 Hz; D) on the type of the applied emulsifying salt (dipotassium hydrogenphosphate [DSP], tetrapotassium diphosphate [TKPP], pentapotassium triphosphate [KTPP]) during a 60-d storage period at $6 \pm 2 \degree C$ [n = 6; the results were expressed as means (columns) and standard deviations (bars); processed cheeses were sampled after 1 (black), 14 (silver), 30 (dark-gray), 60 (light-gray) d of storage].

reflection, using a black (L* = 0) and a white (L* = 100) reference standard. The value L* represents the sample luminosity, varying from 0 (black) to 100 (white); the value a* represents color, varying from red (+) to green (-); and the value b* also represents color, varying from yellow (+) to blue (-) (Chen and Liu, 2012). A 10-mm quartz cuvette was used for the measurements. Each sample was measured in triplicate (n = 3).

Differential Scanning Calorimetry of PC

Thermal analysis was performed with a differential scanning calorimetry (**DSC**) 250 Discovery (TA Instruments) with Tzero patented technology. Calibration was performed by indium standard with peak melting temperature 156.60°C and heat of melting 28.71 J g⁻¹. Each sample (10.0 ± 1.1) mg was filled in Tzero aluminum pin hole hermetic pans (with hole 1 mm), and an empty aluminum pan was used as a reference. Thermograms were obtained by scanning from 25°C to -50°C using cooling ramp of 10°C min⁻¹, subsequently isothermal step at -50°C for 1 min, and following heating cycle in temperature

range -50 to 80° C at a heating rate of 5° C min⁻¹. The measurement was carried out under nitrogen atmosphere at flow rate 50 mL min⁻¹. The state of water in PC was evaluated by parameters of ice melting temperature and relevant enthalpy of fusion (ΔH_{fus} ; Dalmazzone et al., 2009; Tylewicz et al., 2016). For the cooling cycle, the freezing point was evaluated by the following parameters: water crystallization temperature (T_{cryst}) and freezing enthalpy ($\Delta \mathbf{H}_{crvst}$). For the heating cycle: endothermal onset (T_{onset}) , melting peak temperature $(T_{p,m})$, and ΔH_{fus} were evaluated. Enthalpy was calculated as integral area below thermograms and expressed as normalized enthalpy (J g^{-1}). Freezable water (i.e., freezable free and freezable bound water) content in PC was calculated according to Equation [2] as the ratio of ΔH_{fus} of sample to enthalpy change of pure water (307.5 J g⁻¹) measured in the same conditions, instead of standardized heat of fusion value $(\Delta H^0_{m, H2O} = 333.5 \text{ J g}^{-1})$ for pure water.

$$W_{f,s}\left(\%\right) = \frac{\Delta \mathbf{H}_{fus}}{\Delta \mathbf{H}_{m,H2O}^{0}} \times 100, \qquad [2]$$

where $W_{f,s}$ is freezable free and freezable bound water content (Yudianti et al., 2009; Yang and Mather, 2022).

Sensory Analysis of PC

Sensory evaluation of the PC samples was realized with 16 trained assessors in accordance to ISO 8586 (ISO, 2012) in a sensory laboratory equipped with sensory booths under standard lighting conditions (at a controlled temperature of $20 \pm 2^{\circ}$ C) in accordance to ISO 8589 (ISO, 2007). PC samples were served at the refrigeration temperature of $6 \pm 2^{\circ}$ C on white plates coded with 3-digit numbers. To avoid carryover effects, water and crackers were provided to rinse the mouth between the evaluation of the PC samples. The evaluation used a 5-point hedonic scales (1: excellent, 3: good, 5: unacceptable) for appearance, hardness, and spreadability, (1: negligible, 3: medium, and 5: excessive) for bitterness and off-flavors. The overall evaluation of the PC samples was assessed using a 7-point hedonic scale (1: extremely good, 7: very bad). Terms for intensity scales were used to describe each point of the used 5-point and 7-point scales.

Statistical Analysis

The data obtained were evaluated for normal distribution (Shapiro-Wilk test; the significance level was 0.05; Minitab 16 software; Minitab Ltd., Coventry, UK). The Shapiro-Wilk test is recommended on the basis of ISO 5479 (ISO, 1997) for the normal distribution testing of a small amount of data. However, the use of typical parametrical tests was rejected because the normal distribution was not acceptable for all data (P < 0.05). Therefore, the data were analyzed using a nonparametric analysis of the variance of Kruskal-Wallis and Wilcoxon tests (Minitab 16 software; Minitab Ltd., Coventry, UK), with the significance level set at 0.05.

RESULTS AND DISCUSSION

Proximate Analysis and Water Activity of the PC

The results of proximate analysis of the developed PC samples after 1 d of storage is summarized in Table 2. The obtained data show that no significant (P > 0.05) changes in DM, FDM, and protein content were observed in the PC samples. Additionally, a slight decrease in the pH (Figure 1A) was observed in relation to the increasing storage time for all model PC samples, regardless of ES type applied. The current decreasing trend in the pH of the samples might be explained by more intensive release of H⁺ (from the molecules of ES) into the melt. The results are in accordance to that previously reported by Khetra

et al. (2015). The pH value can significantly influence several physicochemical changes: peptization, dispersion, hydration and swelling of proteins, emulsification and fat stabilization or pH modification (Guinee et al., 2004; Chen and Liu, 2012; Salek et al., 2015; Weiserová et al., 2011). However, throughout the storage period, the PC samples showed significantly (P < 0.05) higher pH values (Figure 1A) than the reported optimal pH values (5.5–5.8) for PC spreads (Buňka et al., 2014). A possible explanation for the differences in the pH values is the buffering capacity of the ES applied (Salek et al., 2015). The measured pH values for the individually applied potassium-based ES ranged (1) from 6.04 ± 0.01 to 6.14 \pm 0.01 for DKP; (2) from 6.26 \pm 0.01 to 6.34 \pm 0.01 for TKPP); and (3) from 5.81 ± 0.01 to 5.96 ± 0.01 for KTPP. Similar pH values for PC with potassium-based ES were published in the study of Mozuraityte et al. (2019).

The observed aw values of PC manufactured with individual potassium-based ES were (1) from 0.984 \pm 0.001 (1 d) to 0.979 ± 0.002 (60 d) for the DKP sample; (2) from 0.984 ± 0.001 (1 d) to 0.979 ± 0.002 (60 d) for the TKPP sample; and (3) from 0.976 ± 0.001 (1 d) to 0.970 ± 0.002 (60 d) for the KTPP sample. Thus, the significant changes in a_w values were not observed for the tested PC model samples in relation to applied ES (P > 0.05). During the 60-d storage time a slight decrease in the a_w values was observed, however these findings were not statistically significant (P > 0.05). Furthermore, PC samples presented higher aw values than the lower limit a_w value of 0.910. Hence, the optimum value of a_w for the growth of most microorganisms is $a_w > 0.910$; therefore, this parameter is important in defining the risk of microbial growth in foods (Fernández-Salguero et al., 1993).

Dynamic Oscillatory Rheology of PC

Figure 2 illustrates the dependence of the storage (G')and loss (G") moduli of PC samples on frequency (in range of 0.1-100.0 Hz) manufactured with the individual addition of potassium-based ES with different phosphate chain lengths (DKP, TKPP and KTPP) after 1, 14, 30 and 60 d of storage (at $6 \pm 2^{\circ}$ C). All model PC samples exhibited a more elastic character (G' > G'') over the entire tested frequency range. Additionally, similar results were reported by Dularia et al. (2023). Lu et al. (2007) reported that if the G' values are too low; the main cause would be a poorly chosen concentration of ES. Thus, this was not confirmed in the current study and the concentration of 2% wt/wt of ES was also used in the study of Chavhan et al. (2015). Moreover, the results indicate that each individually applied potassium-based ES has a unique effect on the viscoelastic properties of PC (P < 0.05). In general, as the number of phosphate units increases, the values of G' and G" raised, indicating an increase in the



Figure 2. The dependence of storage (G': closed symbols) and loss (G": open symbols) moduli of the processed cheese samples manufactured with different types of emulsifying salts (dipotassium hydrogenphosphate [K₂HPO₄; DKP; circle], tetrapotassium diphosphate [K₄P₂O₇; TKPP; triangle], pentapotassium triphosphate [K₅P₃O₁₀; KTPP; square], on the frequency [range of 0.1–100.0] after 1 d [A], 14 d [B], 30 d [C], 60 d [D] of storage [6 ± 1°C; n = 6]).

rigidity of PC. The latter statement was confirmed in our study, as well as in other studies previously reported by Buňka et al. (2013b) and Nagyová et al. (2014). However, the above-mentioned studies examined the effect of sodium-based ES with different chain lengths (disodium phosphate, tetrasodium diphosphate, and 5 sodium salts of polyphosphate with different mean length [n \approx 5, 9, 13, 20, and 28]). The effect of potassium-based ES on the viscoelastic properties of PC was investigated by Hoffmann et al. (2012). Particularly, in the latter work slightly higher values for G' compared with that of our study were reported, whereas the G" values were similar.

To characterize PC viscoelastic properties during storage, the complex modulus (G*) of elasticity and tan δ values were used (Figure 1C and 1D). It was observed that the G* and thus the rigidity of all PC samples increased proportionally with the prolonging of the storage time (up to 60 d). Moreover, the rise of the rigidity was proved by the rise in G' and G" values (P < 0.05; Figure 2). The later phenomena could be explained by the pH value of the system and the potential hydrolysis

of polyphosphates, which can significantly affect the viscoelastic properties of PC (P < 0.05; Sádlíková et al., 2010). The highest G* value during 60 d of storage was observed in the PC samples manufactured with the addition of KTPP (P < 0.05). These higher G* values can be explained by more intense interactions in the PC samples such as hydrogen or disulfide bridges, calcium bridges, hydrophobic interactions, or electrostatic interactions. These interactions contribute to the formation of a "denser" final PC structure, which causes a higher rigidity in the product. In addition, the tan δ values confirmed the elastic character of the PC (tan $\delta < 1$). The tan δ values showed a predominantly viscous behavior (tan $\delta > 1$ or G'' > G'). According to Dimitreli and Thomareis (2007) the higher the tan δ value, the more viscous-like PC character should be expected. Moreover, the tan δ of all PC during the 60d storage period decreased (P < 0.05; Figure 1D). In general, the above-mentioned results are in accordance with that previously reported by Salek et al. (2016; 2017; 2020), who tested sodium-based ES. On the whole, it could be concluded that that each (potassium-based ES or sodium-based ES) has a unique effect on the viscoelastic properties of PC.

TPA of PC

Values of PC hardness, cohesiveness, and relative adhesiveness were obtained by TPA. The development of PC hardness, depending on the type of potassium-based ES (DKP, TKPP, KTPP) used and on the length of storage (at $6^{\circ}C \pm 2^{\circ}C$) is interpreted in Figure 1B. It was found that the type of ES applied significantly influenced the hardness of PC. The values of hardness increased with the increasing chain length of phosphate ES applied, in the following order: DKP < TKPP < KTPP (P < 0.05), hence, the samples prepared with KTPP were the hardest (P < 0.05). The same trend was observed by Weiserová et al. (2011), Buňka et al. (2013b), Nagyová et al. (2014) and Salek et al. (2015; 2016) who applied sodiumbased ES (with the same phosphate chain length). The above-mentioned authors reported that as the number of phosphate units in the ES increases, the hardness of the PC generally increases (monophosphates < diphosphates < triphosphates < short polyphosphates [<10phosphorus atoms per molecule] < long polyphosphates [>10 phosphorus atoms per molecule]). In general, as the chain length of sodium-based ES, the ion exchange and dispersion ability of the casein fractions increases. This claim was further supported by studies of Buňka et al. (2012, 2013a) and Nagyová et al. (2014). The lower values of hardness measured for DKP and TKPP samples (compared with KTPP) can also be explained by a change in the pH of the system. For PC with a relatively higher pH value (6.5-6.7), a reduction in the intensity of electrostatic interactions can be expected, leading to a weakening of the gel to produce a product with an excessively soft consistency. Conversely, PC with a lower pH (5.5-5.2) close to isoelectric point of the caseins present results in a creation of a greatly stiff product (even crumbly character; Buňka et al., 2014; Nagyová et al., 2014).

The lowest values of hardness of all model PC samples were measured after 1 d of storage, with increasing storage length a gradual increase in hardness of PC samples was observed (P < 0.05). Thus, the maximum hardness values were measured for the samples with KTPP addition at d 60 of storage (P < 0.05). In addition, similar results were previously reported by Awad et al. (2002), Salek et al. (2016, 2017, 2020), and Weiserová et al. (2011). However, it should be mentioned that the latter authors examined the effect of sodium-based ES on PC textural properties. The increasing trend in hardness during storage can be explained by hydrolysis of the ES used with more than 2 phosphorus atoms per molecule (Weiserová et al., 2011; Salek et al., 2020). According to Chavhan et al. (2015) factors causing higher values



Figure 3. The dependence of coefficient of friction (CoF) on the sliding speed (mm s⁻¹; Stribeck curves) of the processed cheese samples manufactured with different types of emulsifying salts (dipotassium hydrogenphosphate [K₂HPO₄; DKP; circle], tetrapotassium diphosphate [K₄P₂O₇; TKPP; triangle], pentapotassium triphosphate [K₅P₃O₁₀; KTPP; square]). The presented curves are representative of the average of triplicate experiments (n = 3). Values of minimum CoF (CoF_{min}) and initial CoF (CoF_i) were obtained from the graph.

of hardness in PC, include decrease in the pH, possible changes in ES binding leading to changes in dissociative properties, or changes in crystallization forms of milk fat. The detection of lower hardness values in PC samples with the addition of DKP and TKPP can be explained by the change in the pH of the system. Consequently, the bidirectional distancing from the pH optimum (5.5–5.8) leads to a decrease in the ion-exchange capacity along with a decrease in the intensity of casein dispersion, which leads to less dense crosslinking of caseins in the cheese matrix (Mizuno and Lucey, 2005, 2007; Nagyová, et al., 2014; Salek et al., 2015).

Moreover, values of relative adhesiveness and cohesiveness were also obtained (data not presented). During the 60-d storage period, a slight increase in cohesiveness values was observed for all PC samples, which ranged from 0.318 to 0.731 (P > 0.05). Relative adhesiveness values for specific potassium-based ES varied depending on the length of storage: DKP from 0.99 (1 d) to 0.44 (60 d); TKPP from 1.15 (1 d) to 0.38 (60 d); KTPP from 1.72 (1 d) to 0.38 (60 d). In general, the relative adhesiveness values of the products decreased with increasing storage time (P > 0.05). The results are in accordance to that regarding of sodium-based ES, previously reported by Buňka et al. (2013a), Weiserová et al. (2011), and Salek et al. (2015). Moreover, it could be stated that the values or relative adhesiveness and cohesiveness are influenced by the chain length of the applied ES (sodium-based or

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	Coolin	g cycle	Heating cycle				
Processed cheese	T _{p,cryst} (°C)	$\begin{array}{c} \Delta H_{cryst} \\ (J g^{-1}) \end{array}$	T _{onset} (°C)	T _{pm} (°C)	ΔH_{fus} (J g ⁻¹)	W _{f,s} (%)	
DKP TKPP KTPP	$\begin{array}{c} -18.50\pm0.36^{a} \\ -20.62\pm0.71^{b} \\ -23.42\pm1.06^{c} \end{array}$	$\begin{array}{c} 76.90 \pm 1.28^a \\ 69.68 \pm 1.31^b \\ 49.14 \pm 1.87^c \end{array}$	$\begin{array}{c} -7.50 \pm 0.31a \\ -7.84 \pm 0.68^{b} \\ -8.13 \pm 0.38^{c} \end{array}$	$\begin{array}{c} -2.39\pm 0.21^{a} \\ -3.06\pm 0.23^{b} \\ -4.49\pm 0.21^{c} \end{array}$	$\begin{array}{c} 84.89 \pm 1.6^{a} \\ 72.28 \pm 2.02^{b} \\ 55.13 \pm 1.59^{c} \end{array}$	$\begin{array}{c} 27.63 \pm 0.48^{a} \\ 23.52 \pm 0.60^{b} \\ 17.94 \pm 0.48^{c} \end{array}$	

Table 3. Differential scanning calorimetry parameters of processed cheese manufactured with different potassium-based emulsifying salts after 30 d of storage ($6 \pm 1^{\circ}$ C); results were expressed as mean \pm SD (n = 6)¹

^{a-c}Mean values within a column (the difference between emulsifying salt types, comparing the same storage time) followed by different superscript letters statistically differ (P < 0.05).

 ${}^{1}T_{p,cryst}$ = water crystallization peak temperature; ΔH_{cryst} = freezing enthalpy; T_{onset} = onset melting temperature; $T_{p,m}$ = ice melting peak temperature; ΔH_{fus} = melting fusion enthalpy; $W_{f,s}$ = freezable free and freezable bound water content.

potassium-based) and also by the prolonging of the storage time.

Tribological Analysis of PC

Friction curves (or Stribeck curves) were obtained by plotting CoF as a function of sliding speed as is presented in Figure 3. In particular, the Stribeck curves can be divided into 3 different regimens, the boundary, the mixed and the hydrodynamic regimens (Ningtyas et al., 2017). As shown in Figure 3, no significant differences in lubrication properties were observed in the tested PC systems with divergent potassium-based ES (P > 0.05). Moreover, lower CoF was observed in the PC sample with KTPP addition in comparison with the PC samples in which DKP and TKPP were used. For all PC samples, the CoF values changed significantly with the increasing sliding speed (P > 0.05). From the results, it can be seen that the curves overlapped each other for PC samples with DKP and TKPP at low speed (>50 mm s^{-1}) and medium speed $(>100 \text{ mm s}^{-1})$. In contrast, in the high-speed regimen $(130-180 \text{ mm s}^{-1})$, the PC sample with KTPP overlapped with the PC sample with TKPP. In particular, the overlapping of the samples reflects their similar gel structure and viscoelastic properties. In addition, a progressive decline in CoF from CoF_i to CoF_{min} was observed. According to (Schädle et al., 2022) the lubrication characteristics of PC in the boundary regimen are governed by the interaction of the structure units in the network, and higher CoF values are due to higher degrees of interaction. In general, as the sliding speed increased, all PC samples showed a gradual decrease in CoF until the mixed regimen. The latter phenomenon could be explained probably by the fact that in the gap between the surface and the food no particles driven up (Godoi et al., 2017). After CoF_i point and with a gradual increase in the sliding speed an increase in the CoF occurred. The latter CoF increase might be explained by the virtue of both thin layer and hydrodynamic lubrication (Nguyen et al., 2017). Subsequently, again a decrease in the samples

CoF was identified, which might be probably attributed by the PC structure breaking down, due to high slidding speed (Nguyen et al., 2017). Although we observed no significant difference (P > 0.05) in CoF_i between the PC samples with DKP, TKPP, the KTPP sample exhibited the lower CoF_{min}. The highest CoF_i value was found for the PC sample with TKPP addition. Furthermore, in the sliding speed range of 2 to 200 mm/s (the suggested speed range to describe mouth-like conditions during consumption of food) the PC samples CoF development followed almost the same pattern (Schädle et al., 2022). Generally, the obtained tribological results showed that the type of potassium-based ES had no significant effect on the lubrication properties of the PC (P > 0.05). Overall, by understanding the tribological properties of PC, manufacturers can optimize ingredients formulation and manufacturing process to develop PC products that will meet consumer expectations for flavor and mouthfeel.

Determination of Potassium, Sodium, Calcium, and Magnesium Content of PC

The results of sodium, potassium, magnesium, and calcium content determination in PC are shown in Table 2. The lowest sodium content was detected in the PC sample produced with DKP addition. In contrast, the highest sodium and as well as potassium level among all the PC tested was observed in the PC sample with TKPP addition (P < 0.05). The sodium content of commercial PC products was investigated by Agarwal et al. (2011). The above-mentioned authors stated, that the sodium content in PC ranged from 7,500 to 15,200 mg/kg. In addition, Johnson et al. (2009) produced PC with reduced sodium content by applying DKP and the final products contained 6,140 mg/kg of sodium. Moreover, the authors stated that the sodium content of reduced-salt level PC should be in the range of 4,800 to 12,000 mg/kg (Johnson et al., 2009). Our results agree with the latter statement. In contrast, the DKP PC samples contained the highest amount of magnesium (P < 0.05). Furthermore,

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Processed cheese	Time (d)	Appearance	Hardness	Spreadability	Bitterness	Off-flavor	Overall rating
DKP	1	1 ^{a,A}	2 ^{a,A}	3 ^{a,A}	1 ^{a,A}	1 ^{a,A}	1 ^{a,A}
	14	1 ^{a,A}	3 ^{b,A}	$3^{a,A}$	1 ^{a,A}	1 ^{a,A}	1 ^{a,A}
	30	1 ^{a,A}	4 ^{c,A}	3 ^{a,A}	1 ^{a,A}	1 ^{a,A}	1 ^{a,A}
	60	1 ^{a,A}	4 ^{c,A}	4 ^{b,A}	1 ^{a,A}	1 ^{a,A}	1 ^{a,A}
ТКРР	1	1 ^{a,A}	$2^{a,A}$	3 ^{a,A}	1 ^{a,A}	1 ^{a,A}	1 ^{a,A}
	14	1 ^{a,A}	$\overline{3}^{b,A}$	3 ^{a,A}	1 ^{a,A}	1 ^{a,A}	$2^{b,B}$
	30	1 ^{a,A}	4 ^{c,A}	3 ^{a,A}	1 ^{a,A}	1 ^{a,A}	$\overline{2}^{b,B}$
	60	1 ^{a,A}	4 ^{c,A}	4 ^{b,A}	$2^{b,B}$	$2^{b,B}$	3 ^{c,B}
КТРР	1	1 ^{a,A}	$2^{a,A}$	$2^{a,B}$	1 ^{a,A}	1 ^{a,A}	$2^{a,B}$
	14	1 ^{a,A}	3 ^{b,A}	3 ^{b,A}	1 ^{a,A}	1 ^{a,A}	$\tilde{2}^{a,B}$
	30	1 ^{a,A}	3 ^{b,B}	4 ^{c,B}	1 ^{a,A}	1 ^{a,A}	$\tilde{2}^{a,B}$
	60	1 ^{a,A}	3 ^{b,B}	$4^{c,A}$	$2^{a,B}$	2 ^{b,B}	$\frac{2}{3^{b,B}}$

Table 4. Sensorial attributes of processed cheese samples for appearance, hardness, spreadability, bitterness, off-flavor, and overall rating evaluated by an expert panel (n = 16); values are expressed as the median¹

^{a-c}Median values within a column (the difference between emulsifying salt types, comparing the same storage time) followed by different lowercase letters statistically differ (P < 0.05); the samples manufactured using different emulsifying salt types were evaluated independently.

^{A,B}Median values within a column (the difference between storage time, comparing the same emulsifying salt type) followed by different uppercase letters differ (P < 0.05); the samples manufactured using different emulsifying salt types were evaluated independently.

¹Appearance, hardness, spreadability: 1 = excellent; 3 = good; 5 = unacceptable. Bitterness and off-flavors: 1 = negligible, 3 = medium, 5 = excessive. Overall rating: 1 = extraordinarily good; 7 = extremely bad.

the calcium content of the PC was not influenced by the applied ES (P > 0.05). The contents of magnesium and calcium were similar for all evaluated PC samples (P > 0.05). These macrominerals represent an important group of nutrients in terms of nutritional content and co-determine quality of dairy products. According to Noël et al. (2008) macrominerals such as sodium, potassium, magnesium and calcium are a significant group of milk nutrients which are required by the human body in amounts >100 mg/d for optimal function.

Instrumental Color Measurement of PC

The results of the instrumental analysis of color of PC samples after 1d of storage are shown in Table 2. On the basis of the presented data, model samples of PC could be characterized as light yellow (positive values of b*) with a faint red tint (positive values of a*). The results are in accordance to that previously reported by Kůrová et al. (2022), who used sodium-based ES. The values of a* and b* were influenced by the type of ES ($P \le 0.05$). A decrease in the above-mentioned values was observed with the increase in the phosphate chain length. However, the L* values were not affected by the type of ES applied (P > 0.05). According to Awad et al. (2002), the lightness of PC is influenced by the level of ES used. The abovementioned authors stated that the more soluble proteins within the PC-matrix (as a result of increased level of ES) may result in a more shinny and less dark PC product. In general, it is accepted that the color of food products can greatly influence consumer preferences and therefore, is considered as an important attribute (Kůrová et al., 2022).

DSC of PC

Using DSC measurements of freezing/melting processes, the influence of the type of potassium-based ES on the content of freezable free and bound water, classified by ΔH_{fus} of the melting of freezable water in the samples was investigated (Dalmazzone et al., 2009; Yang and Mather, 2022). From the cooling cycle, a distinct sharp narrow peak was detected in the temperature range from -15 to



Figure 4. Differential scanning calorimetry patterns exhibited the endothermic peak of ice melting for processed cheese samples manufactured with different emulsifying salts (dipotassium hydrogenphosphate [K₂HPO₄; DKP; red dotted line], tetrapotassium diphosphate [K₄P₂O₇; TKPP; black solid line], pentapotassium triphosphate [K₅P₃O₁₀; KTPP; green dashed line]). Processed cheeses were sampled after 30 d of storage at 6 \pm 2°C (n = 6).



Figure 5. Images of the developed model processed cheese samples manufactured with 3 different potassium-based emulsifying salts (dipotassium hydrogenphosphate [K_2 HPO₄; DKP; A], tetrapotassium diphosphate [K_4 P₂O₇; TKPP; B], pentapotassium triphosphate [K_3 P₃O₁₀; KTPP; C]). The computer vision system used included a digital camera (Coolpix P6000, Nikon, Tokyo, Japan) supported by a copy stand, monitor, and computer.

 -25° C, corresponding to the phase transition of water crystallization. The resulting values of DSC measurements (T_{cryst} ; ΔH_{cryst} for the cooling cycle; and T_{onset} , T_{pm} , and ΔH_{fus} for the heating cycle) of PC produced with an individual addition of potassium-based ES are illustrated in Table 3. Moreover, the values of W_{fs} were calculated according to Equation [2] as freezable free and freezable bound water content (Aktas et al., 1997; Tylewicz et al., 2016). The results showed that the type of potassiumbased ES affected the binding of water into the structure of the PC (P < 0.05). This effect manifests as an increase of water binding in the structure as the phosphate chain length of the ES increased. In other words, the amount of freezable free water in the sample was reduced (P <0.05). With this effect, a shift in the melting temperature of frozen water to lower values was observed (T_{onset}, T_{pm}) with increasing number of phosphates units in ES, as can be seen in Table 3. It can be assumed that the type of ES can influence the formation of protein aggregates and fat clusters (Felix da Silva et al., 2021), and thus affect the water binding to the structure. Figure 4 shows the DSC thermograms with peaks representing the freezing water melting temperature. The area of the peak corresponds to the change in $\Delta_{\rm Hfus}$ and $T_{\rm onset}$ to the initial melting temperature of the PC samples and T_{pm} . It can be observed that as the number of phosphate units in the ES increases, the enthalpy of melting of ice decreases and the initial melting temperature decreases (P < 0.05). Tomaszewska-Gras et al. (2019) investigated different types of goat cheese. Consistent with the findings of this study, a decrease in the enthalpy of melting (ΔH_{fus}) was observed with increasing ES chain length (Figure 4). Ice formation at temperatures much lower than 0°C is associated with the phenomenon of supercooling, which is typical for water/oil emulsions (Clausse, 2010), i.e.,

for the PC in our study. Partial water freezing from supercooling in PC was also concluded by Gliguem et al. (2009), who attributed it mainly to the dependence on the hydrophilic components in the cheese samples and the water retention in the microdomains defined by the fat droplets. These phenomena played an important role in the PC evaluated in our study.

Sensory Analysis

The sensorial attributes of PC (appearance, hardness, spreadability, bitterness, off-flavor, and overall rating) manufactured with the addition of potassium-based ES as a function of storage time are depicted in Table 4. The results showed that the addition of potassium-based ES in PC had no significant effect (P > 0.05) on the appearance, on the development of bitterness, and occurrence of off-flavor. In the study of Chavhan et al. (2015) reported that PC samples manufactured with monopotassium phosphate as ES received the lowest scores. According to the above-mentioned authors potassium cations may be associated with elevated sourness and reduced saltiness. Furthermore, on the basis of the overall evaluation, the PC with the addition of DKP was the highest rated product, followed by the PC with TKPP and the PC with KTPP. Moreover, according to the obtained results, all tested PC samples could be characterized as very good to excellent (Figure 5). These findings can be evaluated very positively, as the major concern for the dairy industry in the use of potassium-based ES in PC is the possible development of bitter to chemical-metallic offflavor in PC products (Johnson et al., 2009; Chavhan et al., 2015). In addition, for all tested samples, a gradual increase in the hardness of PC with an increasing storage time was observed, which corresponds to the results

of rheological and TPA (increasing of G* and hardness of PC samples during storage) analyses. Moreover, PC samples manufactured with the addition of DKP and TKPP achieved higher scores in hardness in comparison with KTPP samples. In terms of spreadability, all model samples were assessed as predominantly good spreadable products; however, the spreadability of the samples was evaluated negatively after 30 d storage.

CONCLUSIONS

The effect of different types of potassium-based ES on the physicochemical, viscoelastic, textural, thermal, tribological, and sensory properties of PC was investigated. The type of potassium-based ES used had a different effect on the functional properties of the PC. All PC samples exhibited a more elastic character. The hardness of the PC increased with the increasing length of the phosphate chain of the ES and also with the prolonging of the storage time. Moreover, it was found that with increasing potassium content in PC, the enthalpy of melting of ice gradually decreases thus, water is more bound in the PC sample. In this study, the possibility of using potassium-based ES for the production of PC with decreased sodium content was demonstrated without the negative effect of these compounds on the sensory properties of the products, especially the development of bitterness. The use of potassium-based ES in PC can offer several health benefits and make attractive PC products to consumers looking for clean label products.

NOTES

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Nonstandard abbreviations used: $\Delta H_{fus} = enthalpy$ of fusion; $\Delta H_{cryst} =$ freezing enthalpy; $a_w =$ water activity; CoF = coefficient of friction; CoF_i = initial coefficient of friction; CoF_{min} = minimum coefficient of friction; DKP = dipotassium hydrogenphosphate; DSC = differential scanning calorimetry; ES = emulsifying salts; FDM = fat in DM; KTPP = tetrapotassium diphosphate; PC = processed cheese; T_{cryst} = water crystallization temperature; TKPP = pentapotassium triphosphate; T_{onset} = endothermal onset; TPA = texture profile analysis; $T_{p,m}$ = melting peak temperature.

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