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**Research Article**

# Comparative appraisal of quality parameters of milk lollies prepared from goat, sheep, cow and buffalo milk during frozen storage

**R. Sindhav[1*], K. K. Ahuja[1], T. Hazra[2], A. K. Solanki[1], N. Vachhani[1] and V. Ramani[3]**

[1]Department of Dairy Technology, College of Dairy Science, Kamdhenu University, Amreli - 365 601, Gujarat, India; [2]Department of Dairy Chemistry, College of Dairy Science, Kamdhenu University, Amreli - 365 601, Gujarat, India; [3]Principal and Dean, College of Dairy Science, Kamdhenu University, Amreli - 365 601, Gujarat, India

## Abstract

**This study evaluated the effect of prolonged frozen storage (–18±2°C) on the sensory, physicochemical, functional and microbiological stability of optimized milk lollies prepared from goat, sheep, cow and buffalo milk. A significant ($p<0.05$) decline in sensory attributes was observed during storage across all milk types; however, buffalo and sheep milk lollies exhibited retention of flavour, texture and overall acceptability compared to cow and goat milk lollies. Total solids increased from 25.49–28.07 per cent to 25.70–28.28 per cent, with an increase observed across all four milk type during frozen storage. Titratable acidity, FFA and proteolysis also showed progressive increases, indicating ongoing biochemical changes under frozen conditions. Conversely, melting rate decreased, reflecting enhanced structural rigidity of the frozen matrix. Antioxidant activity declined significantly during storage, although appreciable activity (>13%) was retained after 180 days. Microbiological analysis revealed a slight reduction in aerobic plate count, with absence of coliforms, yeast and molds throughout storage, confirming product safety. The results demonstrated that milk composition significantly influences frozen storage stability, with buffalo and sheep milk lollies showing enhanced physicochemical and sensory stability due to higher total solids and protein content. The study provides valuable insights into species-specific behavior of milk-based frozen systems and supports the utilization of non-bovine milk for the development of stable and acceptable low-fat frozen dairy products.**

**Keywords:** Antioxidant activity, Lipolysis and proteolysis, Milk lollies, Non-bovine milk, Physico-chemical properties

## Highlights

- Optimized milk lollies prepared from goat, sheep, cow and buffalo milk remained microbiologically safe and sensorial acceptable during 180 days of frozen storage.
- Buffalo and sheep milk lollies exhibited superior storage stability, lower melting rate and better retention of sensory quality due to higher total solids and protein content.
- Frozen storage significantly increased acidity, free fatty acids and proteolysis, while antioxidant activity and sensory scores progressively declined in all milk lollies.

## INTRODUCTION

Frozen dairy confections represent structurally complex, multiphase systems composed of ice crystals, air cells, fat globules and a concentrated unfrozen serum phase containing lactose, proteins, minerals and dissolved solids. The stability and quality of such systems depend on physicochemical interactions during freezing, hardening and storage (Murtaza et al., 2004; Gnanaprakasam, 2021). In low-fat frozen products such as milk lollies (≤2.0% fat as per Food Safety and Standards Regulations, India), the lack of a well-developed fat destabilization network increases dependence on total solids, protein interactions and stabilizer functionality to maintain structural stability and sensory quality (McGhee et al., 2015; Bureau of Indian Standards [BIS], 2022).

Non-bovine milk sources, such as goat, sheep and camel milk, due to their unique compositional profiles and functional properties (Núñez et al., 2024; Siddiqui et al., 2024; Moraes et al., 2025). Sheep milk generally has higher total solids and greater casein content (3.0–3.5%), which enhances mix viscosity, decreases the freezable water fraction and improves resistance to ice recrystallization (Siddiqui et al., 2024; Moraes et al., 2025). Goat milk contains smaller fat globules (approximately 2–3 μm), a higher proportion of short- and medium-chain fatty acids (C6–C12) and lower αs1-casein content, leading to reduced buffering

*Corresponding Author, E-mail: rohitsindhav7@gmail.com

capacity, lower viscosity (approximately 12–13 cP) and a comparatively weaker protein–fat matrix (McGhee et al., 2015; Núñez et al., 2024). These compositional distinctions substantially affect freezing behavior, glass transition phenomena and the stability of frozen dairy matrices during storage.

The freezing process induces phase separation, causing water to crystallize as ice and concentrating solutes in the unfrozen phase. During storage at "18±2°C, thermodynamically driven processes such as Ostwald ripening (ice recrystallization), moisture migration and lactose crystallization persist, though at slower rates (Singh et al., 2014; Bekiroğlu & Özdemir, 2020). Ice recrystallization enlarges small ice crystals, increasing mean crystal size (>50 µm) and adversely affecting texture and mouthfeel. Concurrently, sublimation of surface ice (freezer burn) and water redistribution result in an apparent increase in total solids concentration. These alterations are frequently accompanied by protein destabilization due to partial dehydration and altered interactions within the casein micelles, as well as disruption of the milk fat globule membrane (MFGM), which increases susceptibility to lipolysis and oxidation (Deeth & Fitz-Gerald, 1976; Khan et al., 2017).

While sub-zero temperatures effectively inhibit microbial growth, biochemical reactions such as lipid hydrolysis and protein degradation may continue due to residual enzyme activity. Lipolysis, measured as free fatty acids (expressed as % oleic acid), can gradually increase during storage, even in low-fat systems, as a result of structural damage to fat globules and increased enzyme accessibility (Deeth & Fitz-Gerald, 1976). Similarly, proteolysis, typically evaluated by tyrosine release in trichloroacetic acid (TCA)-soluble fractions (µg/mL), indicates ongoing protein breakdown influenced by freeze-induced structural stress and solute concentration in the unfrozen phase (Folin & Ciocalteu, 1927). Additionally, oxidative reactions and the degradation of bioactive compounds contribute to reduced antioxidant activity, which is commonly assessed by DPPH radical scavenging at 520 nm (Khan et al., 2017).

Sensory quality in frozen dairy products is determined by flavor retention, body and texture, melting resistance and visual appearance, all of which are closely associated with microstructural stability (Gnanaprakasam, 2021; Salinas-González et al., 2025). Melting behavior, usually measured as mL/h under controlled conditions (25±1°C), serves as an indicator of structural stability; lower melting rates reflect stronger protein–fat networks and reduced serum separation (McGhee et al., 2015; Bekiroğlu & Özdemir, 2020). In low-fat systems, however, melting resistance depends more on total solids content and stabilizer interactions (such as carboxymethyl cellulose) than on fat destabilization (Murtaza et al., 2004).

Response Surface Methodology (RSM), particularly Central Composite Rotatable Design (CCRD), is commonly used to model the effects of key variables such as milk fat content (0.5–2.0%) and sweetener concentration (15–25%) on multiple response parameters, including sensory attributes, viscosity, melting rate, color (L*, a*, b*), titratable acidity (% lactic acid) and antioxidant activity. Quadratic polynomial models and desirability functions facilitate the identification of optimal conditions by accounting for interaction and curvature effects within the experimental domain (Gnanaprakasam, 2021; Calabrese et al., 2026).

Despite increasing interest in frozen dairy products from alternative milk sources, comparative studies on the long-term storage stability of multi-species milk lollies under controlled frozen conditions are limited. Comprehensive assessments that include sensory, physicochemical, functional and microbiological parameters over extended storage periods (up to 180 days) are particularly scarce (Salinas-González et al., 2025; Núñez et al., 2024). The present investigation was designed to evaluate the effect of prolonged frozen storage (–18±2°C) on the sensory, physicochemical, functional and microbiological stability of optimized milk lollies prepared from goat, sheep, cow and buffalo milk.

## MATERIALS AND METHODS

**Raw materials and reagents:** Fresh milk samples (goat, sheep, cow and buffalo) were procured from local dairy farmers in the Amreli region (Gujarat, India) and transported to the laboratory under refrigerated conditions (≤5°C) in sterilized, food-grade containers to minimize microbial and compositional changes. Whole goat milk powder (Aadvik Foods and Products Pvt. Ltd., India) and skimmed cow milk powder (Patanjali Ayurved Ltd., India) were obtained directly from the respective manufacturers. Analytical-grade sucrose, finely powdered prior to use, was employed as the sweetening agent. Carboxymethyl cellulose (CMC; Merck Specialities Pvt. Ltd., India) was used as a stabilizer. Food-grade synthetic colour (Bush brand) and flavouring agents (IFF) were incorporated as per formulation requirements. Prepared milk lollies were packaged in silver metallised polyester film pouches (60 µm thickness) and stored under controlled frozen conditions (–18±2°C).

**Preparation of milk lollies from different types of milk employing optimized process:** The optimized formulation for preparation of milk lollies from goat, sheep, cow and buffalo milk was obtained using Response Surface Methodology (RSM) based on Central Composite Rotatable Design (CCRD). The optimized formulation for goat milk lollies consisted of 1.50 per cent fat and 21.27 per cent sweetener with a desirability value of 0.938. Sheep milk lollies showed an optimum combination of 1.63 per cent fat and 21.29 per cent sweetener with a desirability value of 0.957. Similarly, cow milk lollies exhibited the highest optimized fat level (1.69%) with 21.06 per cent sweetener and a desirability value of 0.971, whereas buffalo milk lollies were optimized at 1.64 per cent fat and 21.43 per cent sweetener with a desirability value of 0.946. The high desirability values obtained for all formulations indicated good agreement between predicted and experimental responses and confirmed the adequacy of the developed models for optimization of milk lolly formulations.

Fresh goat, sheep, cow and buffalo milk were standardized to approximately 2.0 per cent fat (w/w) through partial cream separation to comply with low-fat frozen confection standards. The standardized milk was subjected to high-heat treatment at 80°C for 5 min using steam jacketed oven, followed by rapid cooling to 7±1°C in order to reduce microbial load and improve mix stability.

Sucrose and carboxymethyl cellulose (CMC) were incorporated into each milk type according to the optimized formulation under continuous agitation to ensure complete dissolution and uniform dispersion of ingredients. Food-grade colour and flavour were added as per formulation requirements and the mixes were stirred thoroughly until a homogeneous consistency was achieved with proper hydration of the stabilizer.

The prepared mixes were filled into pre-sanitized moulds and wooden sticks were inserted centrally. The moulds were subjected to freezing under controlled conditions in the candy machine, followed by hardening in a hardening tunnel to facilitate uniform ice crystal formation and proper development of the frozen matrix. After hardening, the milk lollies were transferred to a deep freezer at –18±2°C for storage to stabilize the frozen structure and improve product integrity.

After hardening, the milk lollies were demoulded and packaged individually in silver metallized polyester film pouches (60 µm thickness). The packaged products were stored at –18±2°C for 180 days and analyzed at regular intervals for sensory, physico-chemical, functional and microbiological characteristics.

**Frozen storage study:** Optimized milk lollies were packaged and stored under controlled frozen conditions at –18±2°C for a period of 180 days. Samples were withdrawn at predetermined intervals of 30 days (0, 30, 60, 90, 120, 150 and 180 days) for analysis. The products were evaluated for physico-chemical, functional, sensory and microbiological attributes to assess the extent of quality changes during prolonged frozen storage.

**Physicochemical analysis:** Titratable acidity (% lactic acid) and total solids were determined as per BIS (2022) using standard titrimetric and gravimetric methods, respectively. Free fatty acids (FFA) were estimated by the extraction–titration method (Deeth & Fitzgerald, 1976) and expressed as per cent oleic acid. Proteolysis was assessed by measuring tyrosine content in TCA-soluble fractions using the Folin–Ciocalteu method, with absorbance recorded at 650 nm and results expressed as µg tyrosine/mL.

**Determination of antioxidant activity**: Antioxidant activity was determined using the DPPH radical scavenging assay following the modified method of Espin et al. (2000). Two hundred milligram of sample was extracted with 3.8 mL ethyl acetate, followed by addition of 1 mL DPPH working solution ($6.09 \times 10^{-5}$ mol/L). After incubation (10 min), absorbance was recorded at 520 nm (UV–Vis spectrophotometer, Shimadzu UV-1900i). Results were expressed as radical scavenging activity (%):

$$\text{DPPH RSA (\%)} = \frac{\text{Absorbtion of control} - \text{Absorpition of sample}}{\text{Absorption of control}} \times 100$$

**Determination of melting rate:** Melting rate was determined using a modified Association of Official Analytical Chemists [AOAC] (1980) method. Fifty-gram sample was placed on a wire mesh (131 pores/in²) at 25±1°C. Melted volume was collected over 30 min and expressed as mL/h.

**Sensory evaluation:** Sensory evaluation was conducted by a trained panel (n=20; faculty and postgraduate students, 22–45 years) at a serving temperature of –13°C. Samples were evaluated for flavour, colour and appearance, body and texture, melting quality and overall acceptability using a 9-point hedonic scale (1=dislike extremely;

9=like extremely) (Solanki, 2025). Samples were coded and presented in randomized order to minimize bias and potable water was provided for palate cleansing between samples. All panellists participated voluntarily after providing informed consent. The study protocol adhered to institutional ethical guidelines for sensory evaluation involving human participants, ensuring confidentiality and non-invasive assessment procedures.

**Microbiological analysis:** Microbial quality was assessed by determining aerobic plate count in accordance with BIS (2012), yeast and mould count in accordance with Indian Standard Institution [ISI] (1969) and coliform count in accordance with BIS (2002). Results were expressed as $\log_{10}$ cfu/mL.

**Statistical analysis:** All experimental results are presented as mean±standard error. Statistical analysis was performed using IBM SPSS Statistics. Analysis of variance (ANOVA) was applied to assess significance ($p<0.05$) and mean differences were compared using Duncan's Multiple Range Test (DMRT).

**Table 1. Proximate composition of the raw milk of goat, sheep, cow and buffalo milk**

| Species | Fat (%) | Total solids (%) | Protein (%) | Lactose (%) |
|---|---|---|---|---|
| Goat Milk | 3.71±0.42$^{d}$ | 12.35±0.25$^{d}$ | 3.67±0.12$^{c}$ | 4.36±0.17$^{c}$ |
| Sheep Milk | 6.63±0.48$^{b}$ | 17.37±0.30$^{b}$ | 5.43±0.09$^{a}$ | 4.57±0.08$^{b}$ |
| Cow Milk | 4.59±0.36$^{c}$ | 13.69±0.43$^{c}$ | 3.87±0.05$^{c}$ | 4.59±0.06$^{b}$ |
| Buffalo Milk | 8.07±0.32$^{a}$ | 18.35±0.20$^{a}$ | 4.38±0.15$^{b}$ | 5.20±0.19$^{a}$ |

Values are mean±SE (n=3); Superscript a, b, c, d indicates significant ($p<0.05$) differences among the species.

## RESULTS

**Proximate composition of goat, sheep, cow and buffalo milk used for milk lollies preparation:** The proximate composition of raw milk obtained from goat, sheep, cow and buffalo, used for the preparation of milk lollies, is presented in Table 1. The composition of raw milk differed significantly ($p<0.05$) among the species with respect to fat, total solids, protein and lactose content. Buffalo milk showed the highest fat content (8.07±0.32%), followed by sheep milk (6.63±0.48%), cow milk (4.59±0.36%) and goat milk (3.71±0.42%). The differences in fat content among all milk species were statistically significant. Total solids content also varied significantly among species, with buffalo milk recording the highest value (18.35±0.20%), followed by sheep milk (17.37±0.30%), cow milk (13.69±0.43%) and goat milk (12.35±0.25%).

Protein content was highest in sheep milk (5.43±0.09%), followed by buffalo milk (4.38±0.15%). Cow milk (3.87±0.05%) and goat milk (3.67±0.12%) showed comparatively lower protein content and were statistically at par with each other. Lactose content was significantly highest in buffalo milk (5.20±0.19%), followed by cow milk (4.59±0.06%) and sheep milk (4.57±0.08%), while goat milk showed the lowest lactose content (4.36±0.17%).

**Changes in sensory scores of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C:** Flavour scores of milk lollies showed a significant ($p<0.05$) decline during 180 days of frozen storage across all milk types. At 0 day, flavour scores for goat, sheep, cow and buffalo milk lollies were 8.11±0.03, 8.37±0.02, 8.30±0.02 and 8.50±0.03, respectively. These scores decreased to 6.83±0.03, 7.20±0.02, 7.15±0.04 and 7.30±0.05, respectively, at 180 days of storage (Table 2). Among the milk types, goat milk lollies showed the highest initial flavour score, but a greater decline was observed during storage. Cow milk lollies

**Table 2. Changes in sensory scores of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C**

| Parameters | Species | Storage period | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 0 Day | 30 Days | 60 Days | 90 Days | 120 Days | 150 Days | 180 Days |
| Flavour | Goat | 8.11±0.03$^{aC}$ | 8.07±0.07$^{aB}$ | 7.52±0.08$^{bB}$ | 7.46±0.10$^{bcB}$ | 7.30±0.08$^{cdB}$ | 7.09±0.05$^{dB}$ | 6.83±0.03$^{eC}$ |
| | Sheep | 8.37±0.02$^{aB}$ | 8.24±0.02$^{aA}$ | 8.00±0.09$^{bA}$ | 7.80±0.02$^{cA}$ | 7.48±0.07$^{dAB}$ | 7.33±0.05$^{deA}$ | 7.20±0.02$^{eAB}$ |
| | Cow | 8.30±0.02$^{aB}$ | 8.19±0.02$^{bAB}$ | 7.91±0.02$^{cA}$ | 7.70±0.07$^{dA}$ | 7.39±0.03$^{eAB}$ | 7.26±0.02$^{fA}$ | 7.15±0.04$^{gB}$ |
| | Buffalo | 8.50±0.03$^{aA}$ | 8.30±0.05$^{bA}$ | 8.02±0.05$^{cA}$ | 7.87±0.02$^{cA}$ | 7.55±0.09$^{dA}$ | 7.37±0.04$^{eA}$ | 7.30±0.05$^{eA}$ |
| Body and Texture | Goat | 8.28±0.03$^{aB}$ | 8.00±0.06$^{bB}$ | 7.67±0.03$^{cB}$ | 7.41±0.08$^{dB}$ | 7.28±0.06$^{deB}$ | 7.17±0.06$^{eB}$ | 6.94±0.03$^{fB}$ |
| | Sheep | 8.39±0.03$^{aAB}$ | 8.15±0.04$^{bAB}$ | 7.94±0.08$^{cA}$ | 7.81±0.02$^{cdA}$ | 7.69±0.05$^{dA}$ | 7.46±0.05$^{eA}$ | 7.37±0.07$^{eA}$ |
| | Cow | 8.35±0.02$^{aAB}$ | 8.09±0.09$^{bAB}$ | 7.87±0.02$^{cAB}$ | 7.74±0.02$^{cA}$ | 7.59±0.05$^{dA}$ | 7.39±0.03$^{eA}$ | 7.30±0.02$^{eA}$ |
| | Buffalo | 8.42±0.05$^{aA}$ | 8.24±0.05$^{bA}$ | 8.00±0.02$^{cA}$ | 7.85±0.02$^{cdA}$ | 7.72±0.06$^{dA}$ | 7.52±0.05$^{eA}$ | 7.43±0.07$^{eA}$ |

**Cont. Table 1.**

Table 1., Cont. ...

| Parameters | Species | Storage period | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 0 Day | 30 Days | 60 Days | 90 Days | 120 Days | 150 Days | 180 Days |
| Colour and Appearance | Goat | 8.41±0.02$^{aBC}$ | 7.98±0.02$^{bB}$ | 7.69±0.07$^{cB}$ | 7.59±0.08$^{cB}$ | 7.26±0.05$^{dB}$ | 7.02±0.05$^{eB}$ | 7.00±0.09$^{eB}$ |
| | Sheep | 8.33±0.03$^{aC}$ | 8.15±0.07$^{bAB}$ | 7.83±0.08$^{cAB}$ | 7.76±0.02$^{cdA}$ | 7.65±0.02$^{dA}$ | 7.48±0.07$^{eA}$ | 7.28±0.03$^{fA}$ |
| | Cow | 8.46±0.02$^{aAB}$ | 8.07±0.07$^{bAB}$ | 7.78±0.03$^{cB}$ | 7.67±0.03$^{cdAB}$ | 7.56±0.06$^{dA}$ | 7.41±0.02$^{eA}$ | 7.20±0.05$^{fA}$ |
| | Buffalo | 8.54±0.05$^{aA}$ | 8.22±0.03$^{bA}$ | 8.02±0.07$^{cA}$ | 7.80±0.02$^{dA}$ | 7.69±0.02$^{dA}$ | 7.52±0.05$^{eA}$ | 7.33±0.03$^{fA}$ |
| Melting Quality | Goat | 7.70±0.07$^{aC}$ | 7.59±0.05$^{abB}$ | 7.46±0.07$^{bcB}$ | 7.35±0.05$^{cC}$ | 7.19±0.04$^{dB}$ | 7.04±0.05$^{deB}$ | 6.91±0.02$^{eB}$ |
| | Sheep | 8.04±0.05$^{aAB}$ | 7.93±0.02$^{abA}$ | 7.85±0.02$^{bA}$ | 7.70±0.02$^{cAB}$ | 7.46±0.05$^{dA}$ | 7.41±0.05$^{dA}$ | 7.35±0.05$^{dA}$ |
| | Cow | 7.94±0.03$^{aB}$ | 7.89±0.03$^{aA}$ | 7.76±0.05$^{bA}$ | 7.61±0.03$^{cB}$ | 7.37±0.04$^{dA}$ | 7.35±0.02$^{dA}$ | 7.28±0.03$^{dA}$ |
| | Buffalo | 8.11±0.03$^{aA}$ | 8.02±0.05$^{aA}$ | 7.91±0.02$^{bA}$ | 7.74±0.02$^{cA}$ | 7.50±0.03$^{dA}$ | 7.44±0.03$^{dA}$ | 7.41±0.05$^{dA}$ |
| Overall Acceptability | Goat | 7.96±0.04$^{aC}$ | 7.46±0.05$^{bB}$ | 7.39±0.03$^{bB}$ | 7.31±0.02$^{bD}$ | 7.15±0.08$^{cB}$ | 6.94±0.06$^{dB}$ | 6.76±0.05$^{eC}$ |
| | Sheep | 8.32±0.04$^{aB}$ | 8.00±0.03$^{bA}$ | 7.78±0.08$^{cA}$ | 7.59±0.02$^{dB}$ | 7.39±0.08$^{eA}$ | 7.28±0.03$^{eA}$ | 7.09±0.02$^{fAB}$ |
| | Cow | 8.33±0.06$^{aB}$ | 7.94±0.03$^{bA}$ | 7.69±0.04$^{cA}$ | 7.52±0.02$^{dC}$ | 7.31±0.05$^{eAB}$ | 7.20±0.05$^{eA}$ | 7.00±0.06$^{fB}$ |
| | Buffalo | 8.48±0.02$^{aA}$ | 8.11±0.08$^{bA}$ | 7.87±0.07$^{cA}$ | 7.67±0.03$^{dA}$ | 7.48±0.05$^{eA}$ | 7.33±0.03$^{efA}$ | 7.20±0.07$^{fA}$ |

Values are mean±SE; superscript in lowercase (a, b, c, d) indicates significant ($p<0.05$) differences for different storage days within the species; superscript in uppercase (A, B, C, D) indicates significant ($p<0.05$) differences for same storage days among different species.

recorded the lowest flavour score at the end of storage. Buffalo and sheep milk lollies retained comparatively better flavour scores during prolonged frozen storage.

Body and texture scores of milk lollies declined significantly ($p<0.05$) during frozen storage across all milk types. At 180 days of storage, buffalo milk lollies recorded the highest body and texture score (7.43±0.07), followed by sheep (7.37±0.07), cow (7.30±0.02) and goat milk lollies (6.94±0.03) (Table 2). The decline in body and texture scores followed a trend similar to flavour deterioration during storage. Buffalo and sheep milk lollies exhibited comparatively better retention of textural quality throughout the storage period. Goat milk lollies showed the greatest reduction in body and texture scores during frozen storage.

Colour and appearance scores of milk lollies decreased significantly ($p<0.05$) during frozen storage across all milk types. The decline was relatively gradual up to 60 days of storage, followed by a more pronounced reduction thereafter. At 180 days, buffalo milk lollies recorded the highest colour and appearance score (7.33±0.03), followed by sheep (7.28±0.03), cow (7.20±0.05) and goat milk lollies (7.00±0.09) (Table 2). Among the milk types, buffalo and sheep milk lollies retained comparatively better visual quality throughout storage, whereas goat milk lollies exhibited the greatest decline. In goat milk lollies, colour and appearance scores decreased from 8.41±0.02 at 0 day to 7.00±0.09 at 180 days of storage.

Melting quality scores of milk lollies declined significantly ($p<0.05$) with increasing storage duration across all milk types. Among the different species, goat milk lollies consistently exhibited the lowest melting quality scores throughout storage, whereas buffalo and sheep milk lollies demonstrated comparatively better melting stability. At 180 days of storage, buffalo and sheep milk lollies maintained melting quality scores above 7.30, indicating better structural stability during frozen storage. In contrast, goat milk lollies showed a more pronounced decline in melting quality scores over the storage period.

Overall acceptability scores of milk lollies decreased significantly ($p<0.05$) with increasing storage duration across all milk types. The decline followed trends similar to those observed for individual sensory attributes. However, all samples remained within the acceptable sensory limit (>6.5) throughout 180 days of frozen storage at −18±2°C. At the end of storage, buffalo milk lollies recorded the highest overall acceptability score (7.20±0.07), followed by sheep (7.09±0.02), cow (7.00±0.06) and goat milk lollies (6.76±0.05) (Table 2). Goat milk lollies exhibited the greatest decline in overall acceptability during storage.

**Changes in total solids contents of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C:** The total solids content of optimized milk lollies prepared from goat, sheep, cow and buffalo milk increased slightly but significantly ($p<0.05$) during 180 days of frozen storage at −18±2°C. Initially, total solids ranged from 25.49 per cent in goat milk lollies to 28.07 per cent in sheep milk lollies, while buffalo and cow milk lollies contained 27.39 per cent and 25.92 per cent, respectively. At the end of storage, total solids increased to 25.70 per cent (goat), 28.28 per cent

(sheep), 26.21 per cent (cow) and 27.61 per cent (buffalo), indicating a gradual increase during storage (Table 3). Among the milk types, sheep milk lollies consistently exhibited the highest total solids throughout storage, followed by buffalo, cow and goat milk lollies.

**Changes in titratable acidity and melting rate of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C:** The titratable acidity of optimized milk lollies prepared from goat, sheep, cow and buffalo milk increased significantly ($p<0.05$) during 180 days of frozen storage at –18±2°C. Initially, acidity ranged from 0.162±0.003 per cent lactic acid in goat milk lollies to 0.196±0.006 per cent in sheep milk lollies, while cow and buffalo milk lollies showed intermediate values. By 180 days, acidity increased to 0.180±0.002 per cent (goat), 0.217±0.001 per cent (sheep), 0.194±0.003 per cent (cow) and 0.215±0.003 per cent (buffalo) (Table 3). Sheep and buffalo milk lollies consistently exhibited higher acidity values throughout storage, whereas goat milk lollies showed the lowest acidity.

The melting rate of optimized milk lollies prepared from goat, sheep, cow and buffalo milk decreased significantly ($p<0.05$) during 180 days of frozen storage at –18±2°C. Initially, melting rates ranged from 83.25±0.29 mL/h in sheep milk lollies to 98.92±0.44 mL/h in goat milk lollies. At the end of storage, melting rates decreased to 80.60±0.38 mL/h (sheep), 84.17±0.33 mL/h (buffalo), 86.60±0.86 mL/h (cow) and 95.33±0.17 mL/h (goat) (Fig. 1). Among the milk types, sheep and buffalo milk lollies exhibited lower melting rates and better melting resistance throughout storage, whereas goat milk lollies consistently showed the highest melting rates.

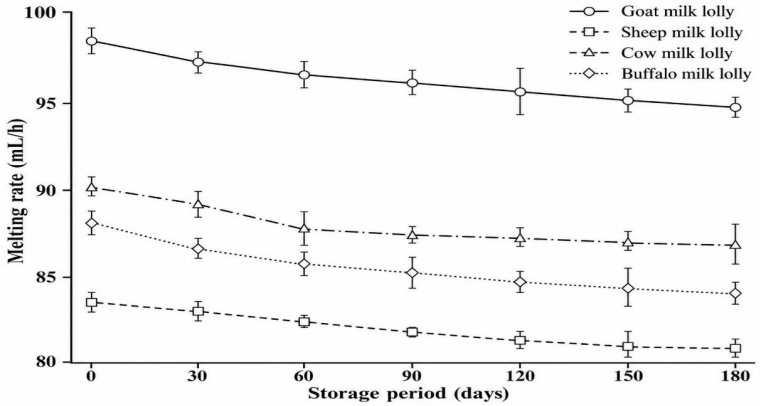



**Fig. 1. Changes in the melting rate (mL/h) of milk lollies from goat, sheep, cow and buffalo milk during storage at –18±2°C**

**Changes in antioxidant activity of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18 ± 2°C:** Antioxidant activity, expressed as DPPH radical scavenging activity (%), decreased significantly ($p<0.05$) during 180 days of frozen storage in milk lollies prepared from goat, sheep, cow and buffalo milk. Initially, antioxidant activity ranged from 13.99±0.50 per cent in goat milk lollies to 17.10±0.30 per cent in sheep milk lollies, while buffalo and cow milk lollies showed intermediate values. By 180 days, antioxidant activity declined to 13.06±0.21 per cent (goat), 16.22±0.30 per cent (sheep), 15.91±0.20 per cent (cow) and 15.46±0.40 per cent (buffalo) (Fig. 2). Among the milk types, sheep and cow milk lollies consistently exhibited higher antioxidant activity throughout storage, whereas goat milk lollies showed the lowest values.

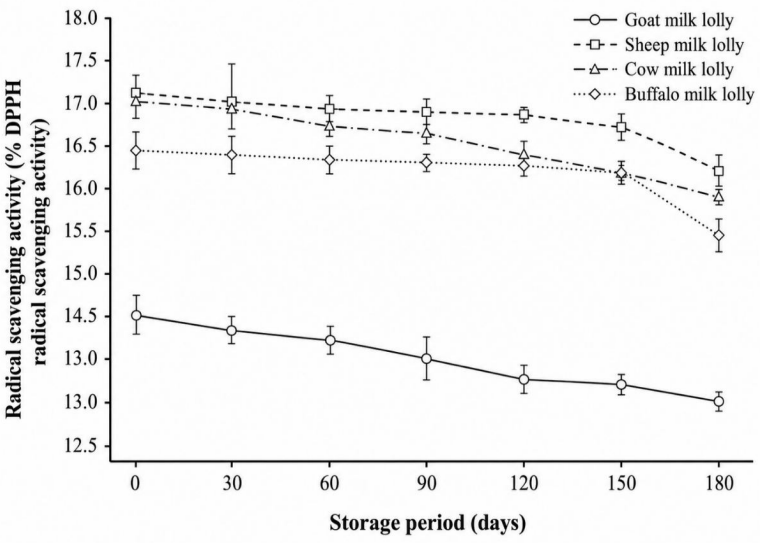



**Fig. 2. Changes in antioxidant activity of milk lollies from goat, sheep, cow and buffalo milk during storage at –18±2°C**

**Changes in free fatty acid content and proteolysis of goat, sheep, cow and buffalo milk lollies during storage at –18±2°C:** FFA content, expressed as % oleic acid, increased significantly ($p<0.05$) during 180 days of frozen storage in milk lollies prepared from different milk types.

**Table 3. Changes in total solid and titratable acidity of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C**

| Parameter | Species | Storage period | | | | | |
|---|---|---|---|---|---|---|---|
| | | 0 Day | 30 Days | 60 Days | 90 Days | 120 Days | 150 Days | 180 Days |
| Total Solid (%) | Goat | 25.49±0.06$^{bD}$ | 25.61±0.04$^{abD}$ | 25.64±0.05$^{abD}$ | 25.66±0.06$^{abD}$ | 25.68±0.04$^{abD}$ | 25.70±0.07$^{aD}$ | 25.70±0.09$^{aD}$ |
| | Sheep | 28.07±0.04$^{bA}$ | 28.13±0.07$^{abA}$ | 28.18±0.07$^{abA}$ | 28.21±0.06$^{abA}$ | 28.24±0.05$^{abA}$ | 28.27±0.04$^{aA}$ | 28.28±0.05$^{aA}$ |
| | Cow | 25.92±0.08$^{bC}$ | 26.07±0.08$^{abC}$ | 26.13±0.07$^{abC}$ | 26.15±0.06$^{abC}$ | 26.18±0.05$^{aC}$ | 26.21±0.10$^{aC}$ | 26.21±0.08$^{aC}$ |
| | Buffalo | 27.39±0.05$^{bB}$ | 27.54±0.05$^{aB}$ | 27.55±0.06$^{aB}$ | 27.57±0.05$^{aB}$ | 27.59±0.04$^{aB}$ | 27.60±0.03$^{aB}$ | 27.61±0.04$^{aB}$ |
| Titratable Acidity (% lactic acid) | Goat | 0.162±0.003$^{dC}$ | 0.165±0.001$^{dC}$ | 0.170±0.001$^{cC}$ | 0.172±0.001$^{cD}$ | 0.174±0.001$^{bcD}$ | 0.177±0.001$^{abD}$ | 0.180±0.002$^{aC}$ |
| | Sheep | 0.196±0.006$^{cA}$ | 0.197±0.005$^{cA}$ | 0.201±0.009$^{bcA}$ | 0.210±0.002$^{abA}$ | 0.213±0.001$^{aA}$ | 0.215±0.001$^{aA}$ | 0.217±0.001$^{aA}$ |
| | Cow | 0.173±0.003$^{eBC}$ | 0.178±0.001$^{deB}$ | 0.181±0.001$^{cdB}$ | 0.183±0.001$^{bcdC}$ | 0.185±0.001$^{bcC}$ | 0.187±0.001$^{bC}$ | 0.194±0.003$^{aB}$ |
| | Buffalo | 0.185±0.005$^{eAB}$ | 0.187±0.002$^{deB}$ | 0.188±0.002$^{cdeB}$ | 0.194±0.001$^{bcdB}$ | 0.197±0.001$^{bcB}$ | 0.201±0.002$^{bB}$ | 0.215±0.003$^{aA}$ |

Values are mean±SE (n=3); superscript in lowercase (a, b, c, d) indicates significant ($p<0.05$) differences for different storage days within the species; superscript in uppercase (A, B, C, D) indicates significant ($p<0.05$) differences for same storage days among different species

**Table 4. Changes in free fatty acid content and proteolysis of goat, sheep, cow and buffalo milk lollies during frozen storage at –18±2°C**

| Parameter | Species | Storage period | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 0 Day | 30 Days | 60 Days | 90 Days | 120 Days | 150 Days | 180 Days |
| Free Fatty Acid (% oleic acid) | Goat | 0.168±0.001$^{eB}$ | 0.169±0.001$^{deC}$ | 0.170±0.001$^{dC}$ | 0.176±0.001$^{cB}$ | 0.177±0.001$^{bcB}$ | 0.179±0.001$^{bB}$ | 0.183±0.002$^{aB}$ |
| | Sheep | 0.173±0.001$^{dA}$ | 0.173±0.001$^{dB}$ | 0.173±0.001$^{dB}$ | 0.180±0.001$^{cA}$ | 0.181±0.001$^{cA}$ | 0.183±0.001$^{bA}$ | 0.188±0.001$^{aA}$ |
| | Cow | 0.175±0.001$^{dA}$ | 0.175±0.001$^{dA}$ | 0.176±0.001$^{dA}$ | 0.180±0.001$^{cA}$ | 0.181±0.001$^{cA}$ | 0.184±0.001$^{bA}$ | 0.187±0.001$^{aA}$ |
| | Buffalo | 0.174±0.001$^{dA}$ | 0.175±0.001$^{dA}$ | 0.175±0.001$^{dA}$ | 0.179±0.001$^{cA}$ | 0.181±0.001$^{cA}$ | 0.183±0.001$^{bA}$ | 0.185±0.001$^{aAB}$ |
| Proteolysis (µg of tyrosine /mL) | Goat | 82.77±0.96$^{cC}$ | 84.73±0.86$^{bcC}$ | 85.17±0.60$^{bcC}$ | 85.35±0.61$^{bcC}$ | 86.05±0.18$^{bC}$ | 124.69±0.71$^{aB}$ | 126.24±1.47$^{aB}$ |
| | Sheep | 88.70±0.35$^{cA}$ | 89.80±0.85$^{bcA}$ | 90.33±0.44$^{bcA}$ | 91.58±0.60$^{bA}$ | 92.14±0.31$^{bA}$ | 131.74±0.94$^{aA}$ | 132.69±1.62$^{aA}$ |
| | Cow | 85.53±0.29$^{dB}$ | 86.70±0.35$^{cdBC}$ | 87.67±0.44$^{cB}$ | 88.17±0.33$^{cB}$ | 88.72±0.22$^{cB}$ | 125.93±1.35$^{bB}$ | 127.91±0.70$^{aB}$ |
| | Buffalo | 87.60±0.87$^{cAB}$ | 88.37±0.91$^{cAB}$ | 90.67±0.88$^{bA}$ | 91.83±0.33$^{bA}$ | 92.54±0.66$^{bA}$ | 134.41±0.44$^{aA}$ | 135.33±0.55$^{aA}$ |

Values are mean±SE (n=3); superscript in lowercase (a, b, c, d) indicates significant ($p<0.05$) differences for different storage days within the species; superscript in uppercase (A, B, C, D) indicates significant ($p<0.05$) differences for same storage days among different species

Initially, FFA values ranged from 0.168±0.001 per cent in goat milk lollies to 0.175±0.001 per cent in cow milk lollies. At the end of storage, FFA values increased to 0.183±0.002 per cent (goat), 0.188±0.001 per cent (sheep), 0.187±0.001 per cent (cow) and 0.185±0.001 per cent (buffalo) (Table 4). Sheep and cow milk lollies showed slightly higher FFA accumulation during storage, whereas goat milk lollies exhibited comparatively lower FFA values during the early storage period.

Proteolysis, expressed as soluble peptide content (µg/mL), increased significantly ($p<0.05$) during 180 days of frozen storage in milk lollies prepared from goat, sheep, cow and buffalo milk. Initially, values ranged from 82.77±0.96 µg/mL in goat milk lollies to 88.70±0.35 µg/mL in sheep milk lollies. Proteolysis increased progressively during storage and reached 126.24±1.47 µg/mL (goat), 132.69±1.62 µg/mL (sheep), 127.91±0.70 µg/mL (cow) and 135.33±0.55 µg/mL (buffalo) at 180 days (Table 4). Buffalo and sheep milk lollies consistently exhibited comparatively higher proteolysis throughout storage.

**Changes in microbiological counts of milk lollies prepared from goat, sheep, cow and buffalo milk during storage at –18±2°C:** Microbiological evaluation of optimized milk lollies prepared from goat, sheep, cow and buffalo milk during 180 days of frozen storage

**Table 5. Changes in microbiological scores of milk lollies prepared from goat, sheep, cow and buffalo milk during frozen storage at –18±2°C**

| Parameter | | Storage period | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Species | 0 Day | 30 Days | 60 Days | 90 Days | 120 Days | 150 Days | 180 Days |
| Aerobic | Goat | 4.37±0.04$^{aC}$ | 4.36±0.03$^{aC}$ | 4.34±0.02$^{aC}$ | 4.33±0.01$^{aC}$ | 4.33±0.01$^{aC}$ | 4.33±0.02$^{aC}$ | 4.33±0.01$^{aC}$ |
| Plate Count | Sheep | 4.58±0.02$^{aB}$ | 4.57±0.01$^{aB}$ | 4.55±0.02$^{aB}$ | 4.54±0.01$^{aB}$ | 4.54±0.01$^{aB}$ | 4.54±0.02$^{aB}$ | 4.53±0.02$^{aB}$ |
| ($\log_{10}$ | Cow | 4.57±0.02$^{aB}$ | 4.56±0.02$^{aB}$ | 4.54±0.02$^{aB}$ | 4.53±0.02$^{aB}$ | 4.52±0.01$^{aB}$ | 4.52±0.01$^{aB}$ | 4.52±0.04$^{aB}$ |
| cfu/mL) | Buffalo | 4.73±0.01$^{aA}$ | 4.72±0.01$^{aA}$ | 4.70±0.01$^{aA}$ | 4.69±0.01$^{aA}$ | 4.69±0.01$^{aA}$ | 4.68±0.04$^{aA}$ | 4.67±0.02$^{aA}$ |

Values are mean±SE (n=3); superscript in lowercase (a, b, c, d) indicates significant ($p<0.05$) differences for different storage days within the species; superscript in uppercase (A, B, C, D) indicates significant ($p<0.05$) differences for same storage days among different species

at –18±2°C demonstrated good microbial stability across all samples. Aerobic plate counts (APC) showed a slight, non-significant decline ($p>0.05$) during storage. Initially, APC values ranged from 4.37±0.04 $\log_{10}$ cfu/mL in goat milk lollies to 4.73±0.01 $\log_{10}$ cfu/mL in buffalo milk lollies, while cow and sheep milk lollies showed intermediate values (Table 5). At the end of storage, APC values marginally decreased to 4.33±0.01 (goat), 4.52±0.04 (cow), 4.53±0.02 (sheep) and 4.67±0.02 $\log_{10}$ cfu/mL (buffalo). Goat milk lollies consistently exhibited the lowest APC values throughout storage. Coliforms, yeast and moulds were not detected in any of the samples during the entire storage period.

## DISCUSSION

**Effect of milk species on raw milk composition:** The observed variation in the proximate composition of goat, sheep, cow and buffalo milk may be attributed to inherent species-specific differences in milk composition. Milk composition is influenced by genetic characteristics of the species, breed, stage of lactation, feeding pattern and physiological status of the animal and environmental conditions. In the present study, buffalo and sheep milk showed comparatively higher fat and total solids contents than cow and goat milk, indicating their richer compositional profile. The higher fat content of buffalo milk is consistent with its known compositional characteristics. Buffalo milk generally contains more fat and total solids than cow and goat milk, which contributes to greater richness, higher viscosity and improved body in dairy products. The present findings are consistent with Thesiya (2024), and Hariyani (2024), who reported significant ($p<0.05$) differences in fat content among milk samples from different species.

Total solids content followed a similar trend, with buffalo and sheep milk showing higher values than cow and goat milk. Higher total solids in buffalo and sheep milk are mainly due to their higher fat, protein, lactose and mineral contents. Such compositional richness is technologically important in frozen dairy products, as higher total solids help improve body, texture and melting resistance. Similar observations were reported by Hariyani (2024), and Kapadiya et al. (2016), who observed wide variation in total solids, protein and lactose content among cow, buffalo and goat milk.

Prolonged frozen storage significantly influenced the sensory, physicochemical, functional and microbiological characteristics of milk lollies prepared from goat, sheep, cow and buffalo milk. Although storage at –18±2°C effectively preserved overall product acceptability for 180 days, progressive quality deterioration was observed across all milk types due to physicochemical and structural modifications occurring within the frozen matrix. These changes are characteristic of frozen dairy systems, where dynamic phenomena such as ice recrystallization, moisture migration, solute concentration and destabilization of protein–fat interactions continue even under sub-zero conditions.

Sensory attributes, including flavour, body and texture, colour and appearance, melting quality and overall acceptability, declined significantly ($p<0.05$) during storage. The reduction in sensory quality may primarily be attributed to cumulative structural deterioration caused by recrystallization of ice crystals, dehydration of the surface layer, weakening of the protein matrix and gradual oxidation of flavour compounds. Similar reductions in sensory quality during frozen storage have been reported in frozen dairy products by Murtaza et al. (2004), Singh et al. (2014), Gnanaprakasam (2021), and Bekiroglu and Ozdemir (2020). However, buffalo and sheep milk lollies consistently demonstrated superior sensory retention compared to cow and goat milk lollies, indicating improved structural stability during frozen storage.

The enhanced sensory stability of buffalo and sheep milk lollies can be attributed to their inherently higher total solids, protein and casein contents, which contribute to increased viscosity and formation of a denser frozen matrix. Increased viscosity restricts molecular mobility and reduces freezable water, thereby limiting ice crystal growth and serum separation during storage. Consequently, these products maintained superior body, texture, appearance and melting resistance throughout the storage period. In contrast, goat milk lollies exhibited comparatively greater sensory deterioration, particularly in flavour and texture. This may be associated with the higher concentration of short- and medium-chain fatty acids characteristic of goat milk, which are more susceptible to oxidative degradation and development of characteristic off-flavours during storage (McGhee et al., 2015). Furthermore, the relatively lower ás1-casein content and weaker protein network of goat milk may have contributed to reduced structural rigidity and increased susceptibility to recrystallization-induced textural defects.

Changes in colour and appearance were mainly associated with surface dehydration, sublimation of ice and formation of larger ice crystals, which reduced surface smoothness and glossiness of the products. Similar observations have been reported in stored frozen desserts by Singh et al. (2014). Melting quality also declined progressively during storage. Nevertheless, buffalo and sheep milk lollies maintained comparatively lower melting rates because their stronger protein–fat matrices and higher total solids reduced serum drainage and improved structural integrity during melting. The observed relationship between melting resistance and matrix stability corroborates earlier findings reported by Murtaza et al. (2004), and, Bekiroglu and Ozdemir (2020).

The gradual increase in total solids observed during storage may be attributed primarily to moisture migration and sublimation, which concentrate non-volatile constituents such as lactose, proteins and minerals within the frozen matrix. Similar increases in total solids during frozen storage have been reported by Singh et al. (2014), and Gnanaprakasam (2021). Sheep and buffalo milk lollies exhibited comparatively higher total solids throughout storage because of the inherent compositional characteristics of these milk types. Higher solids content reduces the proportion of freezable water, thereby improving freeze concentration stability and reducing recrystallization phenomena. This explains the superior storage stability, reduced melting rate and improved sensory retention observed in buffalo and sheep milk lollies. Conversely, goat milk lollies contained comparatively lower total solids, resulting in greater freezable water and increased susceptibility to structural destabilization and faster melting during storage, as similarly reported by McGhee et al. (2015).

Titratable acidity increased gradually throughout storage in all products. Although microbial growth remained negligible under frozen conditions, residual enzymatic activity, limited lactose hydrolysis and release of acidic groups from proteins and phospholipids may have contributed to the observed increase in acidity. The increase may also be partly attributed to an apparent concentration of total solids during frozen storage, as moisture migration and surface sublimation can concentrate proteins, phosphates, citrates and other buffering constituents, thereby increasing the amount of alkali required to reach the titration endpoint (Covacevich & Kosikowski, 1979). Similar trends have been reported in frozen dairy products by Murtaza et al. (2004), Singh et al. (2014) and, Gnanaprakasam (2021). Sheep and buffalo milk lollies exhibited comparatively higher acidity, which may be related to their greater buffering capacity due to higher concentrations of proteins, calcium and phosphate salts. In contrast, goat milk lollies showed relatively lower acidity because of lower casein and mineral content as well as structural differences in casein micelles.

A significant reduction in melting rate was also observed during frozen storage. This reduction may be attributed to progressive freeze concentration, moisture redistribution and strengthening of the frozen matrix due to concentration of solids within the unfrozen phase. Ice recrystallization and reduced serum mobility likely contributed to formation of a denser matrix with lower melt flow characteristics. Similar reductions in melting rate during storage have previously been reported by Murtaza et al. (2004). Among the milk types, sheep and buffalo milk lollies exhibited superior melting resistance because of higher viscosity, greater total solids and stronger protein interactions, whereas goat milk lollies exhibited relatively higher melting rates due to weaker structural organization and increased free water content. Cow milk lollies demonstrated intermediate behaviour, reflecting their moderate compositional and structural properties.

Functional quality parameters also changed significantly during frozen storage. Antioxidant activity decreased progressively in all milk lollies, whereas free fatty acid (FFA) content and proteolysis

increased significantly ($p<0.05$), indicating continuing biochemical modifications within the frozen system despite low-temperature storage. The decline in antioxidant activity may be associated with degradation of antioxidant peptides, oxidation-sensitive vitamins and other bioactive compounds caused by oxygen exposure, freeze-induced stress and moisture migration during storage. Similar reductions in antioxidant activity during frozen storage have been reported by Singh et al. (2014) and Khan et al. (2017). Sheep and cow milk lollies retained comparatively higher antioxidant activity, possibly due to the presence of higher concentrations of bioactive proteins, peptides and minerals capable of free radical scavenging through hydrogen donation and metal chelation mechanisms. Despite the observed decline, appreciable antioxidant activity remained after 180 days, suggesting satisfactory preservation of functional attributes under frozen conditions.

FFA content increased progressively during storage, reflecting gradual lipolysis within the frozen products. Freezing and recrystallization can damage the milk fat globule membrane, thereby increasing accessibility of triglycerides to residual lipolytic enzymes. Similar increases in FFA during frozen storage of dairy products have been reported by McGhee et al. (2015). Sheep and cow milk lollies exhibited comparatively higher FFA accumulation, likely because their higher fat and solids contents provided greater substrate availability for lipolytic reactions. Goat milk lollies initially exhibited lower FFA values, potentially due to their smaller fat globule size and unique membrane composition; however, these differences became less pronounced during prolonged storage. Importantly, FFA levels remained below thresholds associated with detectable rancidity, indicating acceptable oxidative stability of all products during storage.

Proteolysis also increased considerably during storage, indicating progressive degradation of milk proteins under frozen conditions. Although low temperatures inhibit extensive enzymatic activity, freeze concentration effects, dehydration stress and disruption of protein structures within the unfrozen phase may enhance susceptibility of proteins to limited proteolytic breakdown. The increase became more pronounced after prolonged storage, suggesting cumulative structural destabilization within the frozen matrix. Buffalo and sheep milk lollies exhibited comparatively higher proteolysis due to their greater protein and casein contents, which provide larger substrate pools for degradation reactions. Similar findings have been reported in frozen dairy systems by Singh et al. (2014), and Murtaza et al. (2004). Nevertheless, the degree of proteolysis observed did not adversely affect sensory acceptability, indicating that peptide accumulation remained within acceptable limits.

Microbiological evaluation demonstrated excellent microbial stability of all milk lollies during frozen storage. Aerobic plate counts showed slight, non-significant reductions throughout storage. Similar reductions in microbial counts during frozen storage have been reported by Singh et al. (2014). Furthermore, coliforms, yeasts and moulds were absent throughout storage, confirming the effectiveness of heat treatment, hygienic processing conditions and absence of post-processing contamination.

The present study demonstrated that optimized milk lollies prepared from goat, sheep, cow and buffalo milk remained microbiologically safe and sensorially acceptable throughout 180 days of frozen storage at $-18\pm2°C$. However, significant ($p<0.05$) changes in quality attributes were observed during storage, reflecting ongoing physicochemical and biochemical transformations within the frozen matrix. A progressive decline in sensory scores, antioxidant activity and melting quality, along with increases in titratable acidity, free fatty acids and proteolysis, indicated structural destabilization, moisture redistribution and limited enzymatic activity under frozen conditions. Among the milk types, buffalo and sheep milk lollies exhibited superior storage stability, retaining higher sensory acceptability and structural integrity due to their higher total solids and protein content, which contributed to a more stable protein–fat matrix and reduced ice recrystallization effects. In contrast, goat milk lollies showed comparatively faster deterioration, primarily due to lower total solids and a weaker structural network, although they remained within acceptable quality limits. The findings highlight the critical role of milk composition in determining the storage stability of low-fat frozen dairy systems. The study supports the potential of non-bovine milk, particularly sheep and buffalo milk, for the development of high-quality frozen confections with improved shelf stability. These results provide a scientific basis for formulation optimization and industrial application of diversified milk sources in frozen dairy products

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**Conflict of interest:** The authors declare that they have no conflict of interest related to this study.

**Author's contribution:** RS: Carried out the experimental work, sample preparation, data collection, analysis and interpretation of results; TH, KKA: Supervised, designed and monitored the research work regularly and contributed to interpretation of the findings; AKS, NV: Assisted in experimental execution, data compilation and manuscript preparation; VR: Provided overall guidance and administrative support. All authors read, reviewed and approved the final manuscript the findings of this study are included within the article.

**Data availability statement:** The data used to support the findings of this study are included within the article.

**Ethical approval:** This research did not require ethical approval because the milk samples were collected during routine milking procedures only.

**Use of artificial intelligence tools:** During the preparation of this work, the authors did not use any artificial intelligence tools for generation or interpretation of scientific data. The authors reviewed and edited the manuscript as required and take full responsibility for the content of the publication.

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