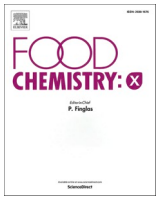
Food Chemistry: X 22 (2024) 101495

Contents lists available at ScienceDirect

Food Chemistry: X

journal homepage: www.sciencedirect.com/journal/food-chemistry-x



Quality characteristics of goat milk powder produced by freeze drying followed by UV-C radiation sterilization

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ARTICLE INFO

Keywords:
Goat milk powder
UV-C radiation
Freeze-dried
Quality characteristics

ABSTRACT

Goat milk was directly freeze-dried into milk powder after freezing and then sterilized using UV-C radiation to produce low-dose, medium-dose and high-dose UV-C radiation sterilized freeze-dried goat milk powder (LGP, MGP and HGP). UV-C sterilization effectively reduced the total bacteria count and coliform bacteria in the goat milk powder while preserving the active proteins, and maintaining the color unchanged. Additionally, LGP, MGP, and HGP all exhibited a moisture content below 5 g/100 g and water activity below 0.5. Upon reconstitution, the milk powder formed uniform and stable emulsion. During accelerated storage tests, the increased Aw did not compromise the microbial quality of milk powder, and there were no significant changes in active proteins as confirmed via SDS-PAGE results. Furthermore, the color parameters (a*, b* and ΔE) showed a strong correlation with hydroxymethyl furfural levels.

1. Introduction

In China, the primary goat milk product is milk powder, which persereves most of the nutrients found in raw milk but with siginificantly reduced moisture content, thereby extending its shelf life (Sanchez, Zhu, Frew, & Kebede, 2020). Spray drying is the most widely used commercial method for producing milk powder (Harizi et al., 2023). However, the thermal treatments required during key stages of production, such as concentration, atomization, droplet-air contact, droplet drying and separation, not only oxidize milk fat and denature milk protein, but also inactivate critical bioactive compounds such as immunoglobulins, growth factors, hormones, oxidative and other sensitive elements (Reddy et al., 2014). Pasteurization, often used before spray drying, is anther form of a heat treatment. Consequently, the utilization of non-thermal processing technologies in the production of milk powder is essential. Freeze-drying, performed at low temperatures, effectively preserves heat-sensitive active ingredients, minimize damage to the original structure and nutrition of the product, and can be used to produce high-quality products.

Our previous research has found that freezing goat milk can assist producers in managing the seasonal nature of goat milk production, low goat milk production and short lactation periods (Yu et al., 2021). Freeze-drying technology enables the direct use of this frozen goat milk as raw material, thus, avoiding the need to thaw it, and facilitates the production of goat milk powder. However, an associated challenges is determining an effective method to sterilize the goat milk powder.

UV-C is one of the few mature non-thermal technologies that can sterilize powders, and has the advantages of fast microbial inactivation, less flavor and nutrient loss, and low energy consumption (Ramos, Esper, & Gonzalez, 2023). UV-C plays the role of killing bacteria by producing photoproducts, making DNA strands unable to replicate, thus leading to cell death (Christen, Lai, Hartmann, Hartmann, & Geddes, 2013). And UV-C has a good germicidal effect on bacteria, fungi, and viruses, and also holds great potential for inactivating bacterial spores (Ansari, Ismail, & Farid, 2019). Matak et al. (2005) demonstrated that UV processing can effectively reduce *Listeria monocytogenes* in goat milk. Cilliers et al. (2014) found that UV-C treatment of whole milk and pasteurization achieved the same effect in reducing the total number of bacteria and mesophilic spores. At the same time, UV-C has less effect on bioactive proteases in milk compared to heat treatment. Studies have shown that, compared with pasteurization, after UV-C sterilization, the activities of catalase and lysozyme in human milk were higher (Martysiak-Żurowska et al., 2017), and the peroxidase in cow milk was not significantly changed (Cilliers et al., 2014). In addition, UV-C treatment may cause oxidation of milk proteins and fats. Milk can absorb ultraviolet rays and produce ozone and nitrogen oxides, leading to fat oxidation and unpleasant odors. The oxidation of proteins is caused by ultraviolet radiation or singlet oxygen, which can affect amino acid

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https://doi.org/10.1016/j.fochx.2024.101495
Received 6 October 2023; Received in revised form 8 May 2024; Accepted 17 May 2024
Available online 19 May 2024

residues such as tryptophan, tyrosine, histidine, and cysteine. However, the degree of protein and fat oxidation is related to UV-C dosage and equipment. Hu et al. (2016) treated milk with static UV-C sterilization equipment and found that there was no significant change in the content of thiobarbituric acid reactive substances (TBARS), while the carbonyl content significantly increased, indicating a certain degree of protein oxidation. Matak et al. (2007) used a commercial UV fluid processor to treat milk and found that the TBARS values of milk increased as UV dose increased.

However, current research on its application in dairy products mainly focuses on liquid milk rather than milk powder. Tedeschi et al. (2023) found that the protein quality, digestibility and biological activity of UV-C treated donkey milk were closer to that of raw milk than those treated with pasteurization. Liu et al. (2020) compared the effects of thermal pasteurization (63 °C for 30 min, 72 °C for 15 s and 85 °C for 5 min), UV-C and ultrasonication on the milk serum proteins, and they found that non-thermal treatments seem to better retain these milk serum proteins, especially for the UV-C treatment.

In this study, we developed a comprehensive non-thermal processing technique to produce high-quality goat milk powder. The process involved directly freeze-dried goat milk into milk powder immediately after freezing, followed by sterilization using varying doses of UV-C radiation. Subsequently, we examined the quality characteristics and storage stability of freeze-dried goat milk powder. This study marks the first application of UV-C radiation for sterilizing goat milk powder, introducing a new approach for the production of high-quality milk powder.

2. Materials and methods

2.1. Sample preparation and experimental design

Fresh goat milk was obtained from a local goat farmer in Xi'an, Shaanxi, China. Samples were obtained by mixing the milk from 5 healthy animals, transported in ice packs to the laboratory, and then frozen to −80 °C overnight, then transferred to a freeze-dryer (FD-1 A-50, Beijing Bomeikang Test Instrument Co., Ltd., Beijing, China) for 48 h to obtain goat milk powder. The temperature was −40 °C, the pressure was 9.5 Pa. Then, unsterilized goat milk powder (UGP) was laid under an ultraviolet lamp, (peak emission at 254 nm, UV-C intensity was 100 $\mu W \cdot cm^{-2}$) (TUV-30 w G30 T8 220 V, Philips, Amsterdam, Netherlands) for 5–10 cm, and sterilized for 15 min, 25 min and 35 min to prepared low-dose UV-C radiation sterilized freeze-dried goat milk powder (LGP, corresponding doses is approximately: 9×10^5 $J \cdot m^{-2}$), medium-dose UV-C radiation sterilized freeze-dried goat milk powder (MGP, corresponding doses is approximately: 1.5×10^6 $J \cdot m^{-2}$) and high-dose UV-C radiation sterilized freeze-dried goat milk powder (HGP, corresponding doses is approximately: 2.1×10^6 $J \cdot m^{-2}$). The UV-C dosage was calculated as follows (Zhang et al., 2021):

UV-C dose ($mJ \cdot cm^{-2}$) = UV-C intensity ($mW \cdot cm^{-2}$) × Exposure time (s).

The obtained powder was stored in sealed polyethylene bags under vacuum conditions for quality analysis and then stored at 35 °C for 42 days with sampling every 7 days for storage stability determination. The experimental design and workflow are shown in Fig. 1.

2.2. Color value determination

Milk color was determined using a spectrophotometer (NS800, Shenzhen Sanenchi Technology Co., Ltd., Shenzhen, China). L* value ranging from 0 to 100, representing black and white 0 and 100, respectively. A positive a* value indicates red, and a negative one suggestes green. Similarly, a positive b* value implies yellow, and a negative one implies blue. The total color difference (ΔE) was calculated relative to FGM as the initial parameter, using Eq. (1):

$$\Delta E = \sqrt{(L - L_0)^2 + (a - a_0)^2 + (b - b_0)^2} \quad (1)$$

where L, a and b are the color parameters of LGP, MGP, and HGP, and L_0, a_0 and b_0 are the color parameters of UGP.

2.3. Particle size and zeta potential determination

The goat milk powder was mixed in distilled water at 1:10 ratio. Subsequently, a laser particle size analyzer (NanoBrook 90 PlusPALS, Brookhaven Instruments, New York, USA) was empplyed to analyze the particle size distribution and Zeta potential of the reconstituted goat milk. The reconstituted goat milk was then diluted 200 times with ultrapure water at room temperature, and an appropriate quantity was taken into the cuvette for measurement. Particle size distribution and Zeta potential analysis were conducted under the following conditions: a real refractive index of 1.59 and refractive index of the fluid (water) of 1.33.

2.4. Microbiological analysis

Total bacteria count (TBC) and coliform count of goat milk powder samples were enumerated by standard plate counting technique according to National Standards of the People's Republic of China (GB 4789.2-2016, 2016 and GB 4789.3-2016, 2016.).

2.5. Analysis of SDS-polyacrilamide gel electrophoresis (SDS-PAGE)

The protein compositions of goat milk powder samples were determined by SDS-PAGE under reducing condition using the method of Ji, Li, Ma, and Li (2017) with modification. The goat milk powder was restored with distilled water at a ratio of 1:10. Reconstituted goat milk were centrifuged in a refrigerated centrifuge at 4 °C and 5727 ×*g* for 10 min to removed the upper milk fat and obtained skimmed milk. Skimmed milk diluting 25 times (32 μL) was mixed with 5× SDS-PAGE sample loading buffer (8 μL). Treated samples were heated at 100 °C for 3 min, then loaded 8 μL onto a 12% SDS-polyacrylamide gel (30% Acryl-Bis at 40%, in 1.5 M Tris-HCl buffer, pH 8.8, for stacking gel and 30% Acryl-Bis at 4.8%, in 1.5 M Tris-HCl buffer, pH 8.8, for separating gel). Molecular mass markers with molecular weight between 11 and 245 kDa was applied to the gel. The gel was run at 80 V first, and then 120 V, using the electrophoresis apparatus (DYY-6C, June 1st Instrument Factory,

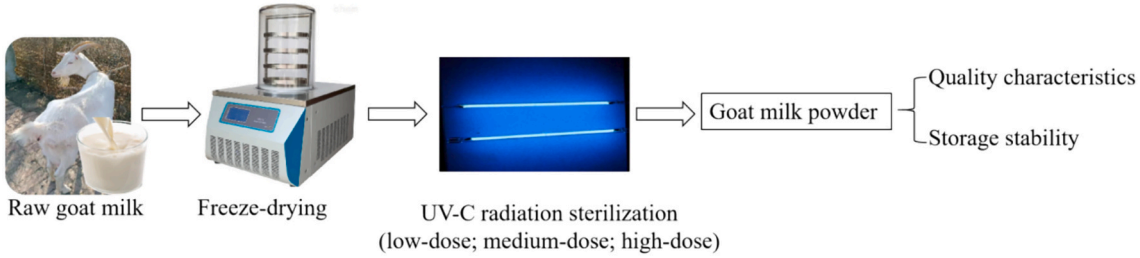

Fig. 1. Experimental design and workflow.

Beijing, China). The protein bands were stained with Coomassie Bright Blue R-250 solution (0.25 g of Coomassie Bright Blue R-250, 40 mL of distilled water, 50 mL of methanol, and 10 mL of glacial acetic acid), and destained with a solution of methanol, glacial acetic acid and distilled water (2:3:5 *v*/v).

2.6. Immunoglobulins and lactoferrin quantification

The goat milk powder was reconstituted with distilled water at 1:10 ratio, followed by centrifugation at 4 °C and 5727 ×*g* for 10 min, and the clear liquid intermediate layer was extracted. Igs (IgG, IgA, IgM) and lactoferrin (LTF) levels were determined using ELISA kits (Shanghai Enzyme-linked Biotechnology Co., Ltd., Shanghai, China) according to provided protocols. Each sample was determined in triplicates.

2.7. Moisture content and water activity

The moisture contents of milk powders were assessed using the direct drying method following the National Standards of the People's Republic of China (GB 5009.3-2016, 2016). Water activity (Aw) of milk powders was measured utilizing a water activity meter (HD-5, Wuxi Huake Instrument Co., Ltd., Wuxi, China).

2.8. Confocal microscopy measurement

The FV1200 confocal scanning laser microscopy (**CSLM**) (Olympus, Tokyo, Japan) was used to observe the structures of reconstituted goat milk samples, following the methodology outlined by Li and Shah (2015). The CLSM was equipped with an inverted microscope and silicon oil objective lens (magnification 150×). Digital images were captured in a tagged image file format. 1 mL of milk samples was mixed with 10 μL of Nile red (1 mg mL^{-1} in ethanol solution) and 10 μL of fluorescein isothiocyanate (FITC, 1 mg mL^{-1} in ethanol solution) allowed to incubate for 30 min. Afterward, 20 μL of stained sample was pipetted onto a glass slide, sealed with a coverslip, and immediately applied to the CLSM immediately. The CLSM observations were conducted in a dark room, with excitation wavelengths set at 534 nm for Nile Red and 488 nm for FITC and emission wavelengths set at 500 to 600 nm and 495 to 559 nm, respectively.

2.9. Determination of the peroxide value

The peroxide value (POV) was determined folllowing the method by Li, Wang, Guo, Shao, and Xu (2019). A 5 g milk powder sample was combined with 5 mL chloroform glacial acetic acid solution (40:60, *v*/v) and 1 mL saturated potassium iodide solution. This mixture was then allowed to react in the dark for 3 min. Subsequently, it was diluted with 50 mL of distilled water, and 1 mL of 1% starch solution (wt/vol) was added. The resulting clear solution was obtained through filtration, and the POV was measured at 585 nm.

2.10. Determination of the thiobarbituric acid value

The thiobarbituric acid (TBA) value was measured according to the methods of Sun (2013) with some adjustments. Intially, goat milk powder was dissolved in distilled water at 1:10 ratio, and 35.2 mL of reconstituted milk (30 °C) was mixed with 2 mL of 40% trichloroacetic acid (*w/v*) and 4 mL of 95% ethanol. After thorough mixing, the mixture was allowed to stand for 15 min and then ifltered. Next, 4 mL of the clarified filtrate was combined with 1.0 mL of 0.1 M TBA solution, and the resulting mixture was incubated in a 60 °C water bath for 60 min. The absorbance was then measured at 538 nm at room temperature.

2.11. Determination of hydroxymethyl furfural

The hydroxymethyl furfural value (HMF) was determined following a method previously reported with cetain modifications (Chávez-Servín, Castellote, & López-Sabater, 2006). Firstly, the goat milk powder was dissolved in distilled water at 1:10 ratio, and 10 mL of reconstituted milk was added with 5 mL of 0.3 M oxalic acid solution and 5 mL of 40% trichloroacetic acid solution (w/v). After shaking, the mixture was allowed to rest for 15 min after shaking and then centrifuged at 4000 ×*g* for 15 min at room temperature. Following centerifigaution, 1 mL of 0.05 M TBA was added to 4 mL of supernatant, and the mixture kept in a water bath at 40 °C for 30 min. The absorbance was measured at 443 nm.

2.12. Statistical analysis

One-way ANOVA was performed using SPSS 24.0 statistical software, and Duncan's multiple range test was used to evaluate the differences between each other. The differences among samples were considered significant with $P < 0.05$.

3. Results and discussion

3.1. The quality characteristics of UV-C radiation sterilized freeze-dried goat milk powder

The initial microbial quality of milk powder is critically linked to its spoilage throughout storage and plays a vital role in maintaining its chemical and sensory properties (Celestino, Iyer, & Roginski, 1997). In this study, the total number of bacteria count and coliform levels in UGP

Table 1
Changes in the characteristics of UV-C radiation sterilized freeze-dried goat milk powder.

Item	UGP	LGP	MGP	HGP
Bacteria count (CFU g^{-1})	$1.62 \times 10^{5\ a}$	1.45×10^{4} b	1.95×10^{4} b	$6 \times 10^{3\ b}$
Coliform (CFU g^{-1})	35	0	0	0
Moisture content (g/100 g)	2.892 ± 0.166^{c}	4.052 ± 0.024^{b}	4.771 ± 0.015^{a}	4.863 ± 0.003^{a}
Water activity (Aw)	0.053 ± 0.017^{b}	0.314 ± 0.007^{a}	0.305 ± 0.015^{a}	0.302 ± 0.005^{a}
IgA (%)	–	99.15 ± 3.21^{a}	99.68 ± 2.58^{a}	98.62 ± 3.14^{a}
IgM (%)	–	99.65 ± 2.61^{a}	99.77 ± 0.20^{a}	98.49 ± 3.42^{a}
IgG (%)	–	86.57 ± 3.01^{c}	99.76 ± 1.14^{a}	93.05 ± 1.81^{b}
LTF (%)	–	95.94 ± 6.92^{a}	99.73 ± 6.34^{a}	98.24 ± 4.17^{a}
Effective diameter (μm)	0.461 ± 0.022^{ab}	0.498 ± 0.04^{a}	0.42 ± 0.008^{b}	0.459 ± 0.054^{ab}
polydispersity index (PDI)	0.285 ± 0.015^{a}	0.234 ± 0.007^{b}	0.249 ± 0.002^{b}	0.179 ± 0.009^{c}
Zeta potential	−43.73 ± 0.59^{c}	−41.58 ± 0.25^{b}	−39.54 ± 1.47^{a}	−41.6 ± 0.66^{b}
L*	87.25 ± 0.21^{b}	86.26 ± 0.1^{c}	88.44 ± 0.08^{a}	86.14 ± 0.31^{c}
a*	−0.58 ± 0.04^{c}	−0.1 ± 0.01^{a}	−0.25 ± 0.03^{b}	−0.24 ± 0.01^{b}
b*	9.58 ± 0.18^{ab}	9.65 ± 0.13^{a}	9.28 ± 0.25^{b}	8.69 ± 0.14^{c}
ΔE	–	1.11 ± 0.09^{b}	1.28 ± 0.03^{b}	1.48 ± 0.23^{a}

[a–c] Different lowercase superscript letters in the same line indicate a significant difference ($P < 0.05$) between the samples.
UGP: unsterilized goat milk powder; LGP: low-dose UV-C radiation sterilized freeze-dried goat milk powder; MGP: medium-dose UV-C radiation sterilized freeze-dried goat milk powder; HGP: high-dose UV-C radiation sterilized freeze-dried goat milk powder. ΔE: total color difference.
IgA (%), IgM (%), IgG (%) and LTF (%): retention of bioactive IgG, IgA, IgM and LTF in freeze-dried goat milk powder after different UV-C radiation sterilized treatments.

were recorded at 1.62 × 10^5 CFU g^{-1} and 35 CFU g^{-1}, respectively, as shown in Table 1. And freeze-dried goat milk powder subjected to various doses of UV-C sterilization confirmed to Chinese standards for milk powder (GB 19644-2010, 2010), with total bacterial counts <2 × 10^4 CFU g^{-1} and coliform counts <10 CFU g^{-1}. These results demonstrate that the efficacy of UV-C radiation as bactericidal method. UV-C treatment have proven capable of achieving a reduction >5-log in *Listeria monocytogenes* in raw goat milk (Matak et al., 2005) and has successfully eliminate *Salmonella* and *E. coli* in a sample of hot pepper, fennel and coriander samples (Hassan et al., 2020). However, its important to note that microorganisms have a certain ability to repair UV-induced damage and can survive post-treatment (Gayán, Condón, & Álvarez, 2014; Salcedo, Andrade, Quiroga, & Nebot, 2007). Moreover, exposure to visible light after UV processing can activate microbial photoactivation (Guerrero-Beltr n & Barbosa-C novas, 2004). Therefore, its crucial to package milk powder immediately after UV-C radiation sterilization to ensure the effectiveness of the treatment and maintain product safety.

The low moisture content and Aw in milk powder are important in inhibiting the growth and reproduction of microorganisms. After UV-C sterilization, the moisture content of freeze-dried goat milk powder was founded to vary by treaments intensity, the order being LGP < MGP < HGP. An increase in UV-C dose extended the exposure time of the milk powder to air, which facilitated to the absorption of environmental moisture, thereby elevating the final product's moisture content. The maximum moisture content recorded for the goat milk powder samples was 4.863 ± 0.003 g 100 g^{-1}, which complies with Chinese standards for milk powder (GB 19644-2010, 2010; Moisture content ≤5%). Its widely, accepted that microbial growth in food is significantly restricted when Aw <0.7, with bacterial, mold and yeast proliferation effectively controlled; a further reduction in Aw <0.5 nearly halts the growth and reproduction of almost all microorganisms (Stevenson et al., 2015). The highest Aw value observed in goat milk powder samples post-UV-C treatment was 0.314, indicating an excellent intiail moisture condtion and water activity level. These factors contribute significantly to the enhanced long-term storability of the freez-dried goat milk goat millk powder following UV-C sterilization.

Immunoglobulins (Igs), together with LTF, lactoperoxidase, and lysozyme, constitute the essential antimicrobial system in milk, contributing to various biological functions inculding regulation, transport, catalysis, and fatty acid binding. After UV-C sterilization, goat milk powder showed a reduction in IgA concentration, sranging from 0.32% to 1.38%. Similarly, the levels of IgM, IgG, and LTF decreased by 0.23% to 1.51%, 0.24% to 13.43%, and 0.27% to 4.06%, respectively. Despite these changes, research by Zhang et al. (2021) indicates that microfiltration has minimal effect on the bioactive proteins in skim milk, which remain largely preserved even after UV-C treatment. In addition, no significant differences were noted in the content of IgA, IgM, and LTF among LGP, MGP, and HGP goat milk powder samples ($P > 0.05$).

In term of appearance, the color and texture of freeze-dried goat milk powder with subjected to varying doses of UV-C sterilization remained relatively consistent. The highest ΔE value observed was 1.48, indicating that the color differnces between sterilized and unsterizled milk powder are visually indistinguishable (Tribst, Falcade, Ribeiro, Júnior, & Oliveira, 2019). Previous studies such as those by Fernandez, Ganan, Guerra, and Hierro (2014) and Keklik, Elik, Salgin, Demirci, and Koçer (2019) have shown that while mild ultraviolet treatment does not alter cheese color, more intense treatments can induce noticeable color changes. Therefore, when utilizing UV-C radiation in food processing, the appropriate dose of UV-C radiation should be controlled in order to maintining the sensory and physicochemical quality.

UV-C sterilization did not significantly alter the effective diameter of the particles in rehydration goat milk powder emulsions ($P > 0.05$). However, there was a notable reduction in the polydispersity index (PDI) ($P < 0.05$), suggesting more uniform distribution of particle sizes. Particulary, the HGP exhibited the smallest PDI value, indicating a narrow and more consistent particle size distribution upon rehydration. Conversely, the absolute value of Zeta potential decreased after rehydration. Notably, the HGP demonstrated the highest absolute Zeta potential value among the three groups, correlating with the minimal PDI value observed in the particle size analysis. The CLMS image (Fig. 2) revealed smaller fat globules in the MGP, consistent with measurements of effective diameter. The HGP samples contained irregular milk fat globules, and slight protein aggregation was observed across all four groups of freeze-dried goat milk powder.

3.2. The storage stability of UV-C radiation sterilized freeze-dried goat milk powder

Temperature plays a vital role in determining the shelf life food during storage. At certain temperature and relative humidity levels, various changes occur in milk powder, including non-enzymatic browning, fat oxidation and the degradation of active ingredients, all of which directly affect its quality. Through accelerated storage test, we can quickly assess the changes in quality and storage stability of milk powder.

During the storage period, the total bacterial count of goat milk powder did not exceed 2 × 10^4 CFU g^{-1}. This is likely atrribuated to the initially low water content and water activity of milk powder, which inhibit microbial growth and reproduction. On the 42 d, the total bacterial count was the lowest in the LGP, followed by MGP, and HGP was the highest, correlating with the trends in water activity (Aw). Over the course of storage, the Aw in the goat milk powder showed a generally upward trend, although it remained below 0.5 for all samples except HGP on 21 d and 42 d (Table 2). This suggests that the increase in Aw does not compromise the microbial quality of UV-C radiation sterilized freeze-dried goat milk powder, as all the samples met the microbial standards set via the Chinese standards for milk powder (GB 19644-2010, 2010).

Li et al. (2019) explored the oxidation stability of 12 kinds of commercially available milk powders during the storage period of 9 months, and noted that b* and L* values are effective indicators of color change in milk powder. In this study, the L* value of MGP and HGP exihibited declining trend during storage. At 42 d, L* value of MGP and HGP decreased significantly by 2.93% and 3.41%, respectively ($P < 0.05$). In contrast, the L* value of LGP did not change significantly in 14–42 d ($P > 0.05$), compared with 0 d. Initially, the a* value of goat milk powder was negative at 0 d and 7 d, but gradually increased to positive values Similarly, the b* value and ΔE value showed an increasing trend throughout the storage period. At the later storage stage, the values of a*, b* and ΔE ranked from large to small as HGP, MGP, and LGP, respectively. Yu, Zheng, and Li (2015) also found that, under various storage conditions, which the first being 25 °C and 50% relative humidity, the second 4 °C and 40%–70% relative humidity, and the third 50 °C and 20%–50% relative humidity, the ΔE of bovine colostrum powder also increased with the storage time.

In the SDS-PAGE image (Fig. 3), we can clearly identify various casein and whey protein in UV-C radiation freeze-dried goat milk powder, including α_{S1}-CN, α_{S2}-CN, β-CN, κ-CN, β-LG and α-LA, etc. The casein bands were apperead wide and dark, indicating their higher content compared to whey proteins. At 14 d, 28 d and 42 d, the bands for β-CN and κ-CN became faint or even disappear, but high molecular weight bands, like LTF, SA and IgG—H, remain clearly visible. Fig. 4 demonstrated the retentions of immunoglobulins and LTF in UV-C radiation sterilized freeze-dried goat milk powder during accelerated storage test. The retention rates for IgA of LGP, MGP, and HGP were all above than 76.84% throughout the entire storage period. The content of IgM and IgG content in MGP did not change significantly from 0 d ($P > 0.05$), maintaining levels above 94%. Husu et al. (1993) also found that immuneglobulin molecules in milk powder seemed to remain active at various storage temperatures (4 °C, 20 °C and 37 °C), with minimal impact on their immune specificity. Contrarily, Yu et al. (2015) reported

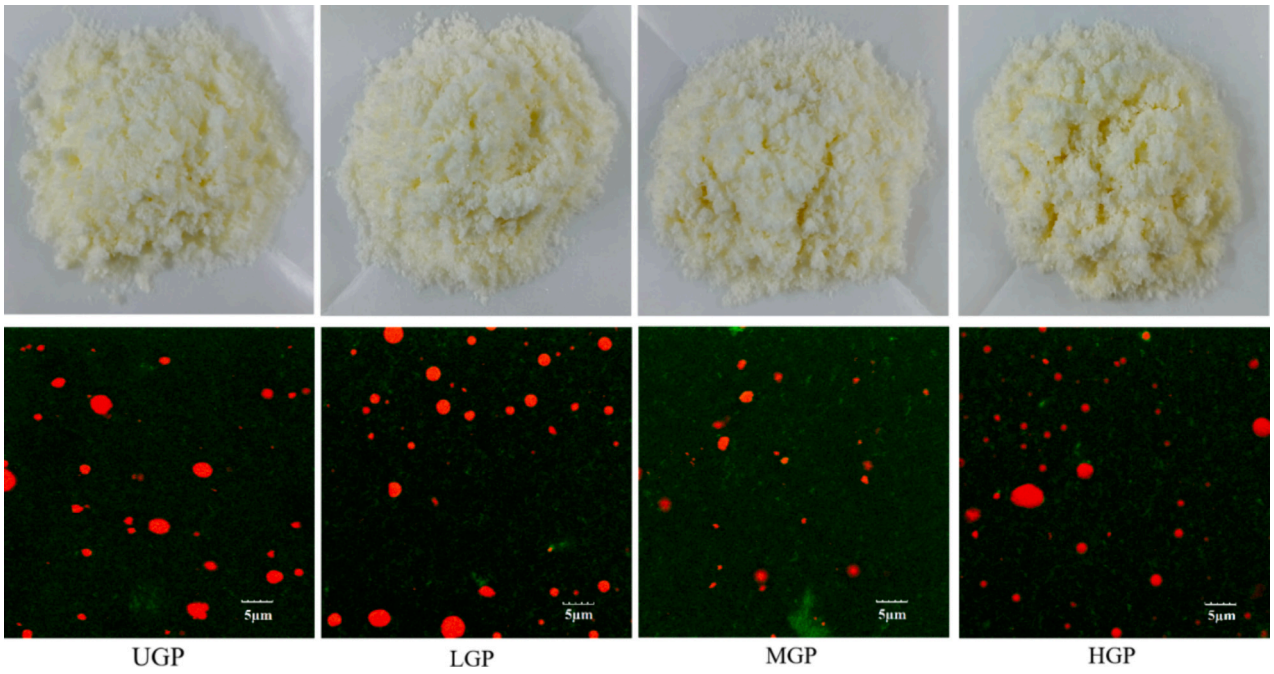

Fig. 2. Appearance and microscopic structures analysis of UV-C radiation sterilized freeze-dried goat milk powder. White bars indicate 5 μm. UGP: unsterilized goat milk powder; LGP: low-dose UV-C radiation sterilized freeze-dried goat milk powder; MGP: medium-dose UV-C radiation sterilized freeze-dried goat milk powder; HGP: high-dose UV-C radiation sterilized freeze-dried goat milk powder.

Table 2
Changes of bacterial count, water activity and color in UV-C radiation sterilized freeze-dried goat milk powder during accelerated storage test.

Storage time (d)	0	7	14	21	28	35	42
Total bacterial count (CFU g^{-1})							
LGP	$14{,}500 \pm 707^{Aa}$	5000 ± 1000^{Ab}	1500 ± 500^{Ac}	1250 ± 150^{Ac}	850 ± 50^{Ac}	900 ± 100^{Ac}	150 ± 50^{Cc}
MGP	$19{,}500 \pm 707^{Aa}$	3000 ± 0^{Bb}	3000 ± 1000^{Ab}	900 ± 200^{Ab}	300 ± 0^{Cb}	300 ± 0^{Bb}	300 ± 40^{Bb}
HGP	6000 ± 1414^{Ba}	1500 ± 500^{Cb}	2500 ± 1500^{Ab}	700 ± 400^{Ab}	550 ± 50^{Bb}	600 ± 300^{ABb}	500 ± 0^{Ab}
Water activity (Aw)							
LGP	0.314 ± 0.007^{Ac}	0.373 ± 0.008^{Ab}	0.363 ± 0.006^{Bb}	0.41 ± 0.008^{Ba}	0.412 ± 0.007^{Ba}	0.407 ± 0.011^{Ba}	0.405 ± 0.012^{Ba}
MGP	0.305 ± 0.015^{Ae}	0.374 ± 0.004^{Ad}	0.369 ± 0.005^{Bc}	0.415 ± 0.008^{Ba}	0.395 ± 0.013^{Bb}	0.415 ± 0.011^{Bb}	0.422 ± 0.005^{Ba}
HGP	0.302 ± 0.005^{Ad}	0.366 ± 0.008^{Ac}	0.392 ± 0.003^{Ac}	0.503 ± 0.001^{Aa}	0.460 ± 0.006^{Ab}	0.456 ± 0.031^{Aa}	0.506 ± 0.017^{Aa}
L*							
LGP	86.26 ± 0.1^{Ba}	85.17 ± 0.11^{Bb}	85.79 ± 0.51^{Aab}	86.07 ± 0.35^{Aab}	85.6 ± 0.74^{Aab}	85.43 ± 0.9^{Aab}	86.3 ± 0.4^{Aab}
MGP	88.44 ± 0.08^{Aa}	84.74 ± 0.34^{Bc}	$84.3 \pm 0.51B^{c}$	85.63 ± 0.9^{Ab}	82.31 ± 0.47^{Cd}	85.44 ± 0.78^{Ab}	85.85 ± 0.35^{Ab}
HGP	86.14 ± 0.31^{Ba}	86.17 ± 0.37^{Aa}	83.32 ± 0.11^{Cb}	81.35 ± 0.83^{Bd}	83.57 ± 0.01^{Bb}	82.41 ± 0.63^{Bc}	83.21 ± 0.27^{Bb}
a*							
LGP	-0.1 ± 0.01^{Ae}	-0.02 ± 0.06^{Ade}	0.03 ± 0.03^{Cd}	0.26 ± 0.03^{Cc}	0.42 ± 0.08^{Cb}	0.62 ± 0.05^{Ca}	0.49 ± 0.07^{Bb}
MGP	-0.25 ± 0.03^{Bd}	-0.21 ± 0.04^{Bd}	0.68 ± 0.1^{Ab}	0.43 ± 0.03^{Bc}	0.75 ± 0.07^{Bb}	1.06 ± 0.01^{Ba}	1.07 ± 0.03^{Aa}
HGP	-0.24 ± 0.01^{Bd}	-0.23 ± 0.09^{Bd}	0.47 ± 0.1^{Bc}	0.56 ± 0.06^{Ac}	1.08 ± 0.14^{Ab}	1.57 ± 0.03^{Aa}	1.18 ± 0.09^{Ab}
b*							
LGP	9.65 ± 0.13^{Ac}	12.03 ± 0.07^{Cb}	12.35 ± 0.24^{Bb}	12.28 ± 1.15^{Bb}	13.64 ± 0.15^{Ca}	14.3 ± 0.41^{Ba}	13.75 ± 0.19^{Ca}
MGP	9.28 ± 0.25^{Bd}	13.26 ± 0.18^{Ac}	14.38 ± 0.39^{Ab}	12.77 ± 0.84^{Bc}	15.01 ± 0.12^{Bab}	14.65 ± 0.36^{Bab}	15.2 ± 0.1^{Ba}
HGP	8.69 ± 0.14^{Cf}	12.91 ± 0.05^{Be}	14.02 ± 0.2^{Ad}	17.39 ± 0.64^{Ac}	16.81 ± 0.18^{Ac}	20.12 ± 0.9^{Aa}	18.73 ± 0.94^{Ab}
ΔE							
LGP	1.11 ± 0.09^{Bd}	3.27 ± 0.10^{Bc}	3.22 ± 0.15^{Bc}	3.12 ± 0.94^{Bc}	4.52 ± 0.42^{Cab}	5.24 ± 0.46^{Ba}	4.42 ± 0.24^{Cb}
MGP	1.28 ± 0.03^{ABe}	4.47 ± 0.26^{Ac}	5.77 ± 0.60^{Ab}	3.84 ± 0.35^{Bd}	7.47 ± 0.27^{Ba}	5.67 ± 0.3^{Bb}	6.03 ± 0.16^{Bb}
HGP	1.48 ± 0.23^{Af}	3.52 ± 0.16^{Be}	6.03 ± 0.12^{Ad}	9.88 ± 0.63^{Ab}	8.28 ± 0.18^{Ac}	11.82 ± 0.55^{Aa}	10.16 ± 0.9^{Ab}

$^{A–C}$ Different superscript uppercase letters indicate a significant difference ($P < 0.05$) between the samples in the same storage time. $^{a-f}$ Different lowercase superscript letters indicate a significant difference ($P < 0.05$) between the different storage times.
LGP: low-dose UV-C radiation sterilized freeze-dried goat milk powder; MGP: medium-dose UV-C radiation sterilized freeze-dried goat milk powder; HGP: high-dose UV-C radiation sterilized freeze-dried goat milk powder. ΔE: total color difference.

that decreased IgG concentration in bovine colostrum powder decreased with storage time, which due to the high temperatures involved in spray drying technology. The LTF retention rates for LGP, MGP, and HGP were all above 78.94%, consistent with the observation of high molecular weight proteins in the SDS-PAGE image. In general, LGP, MGP, and HGP were able to well retain the bioactive proteins during storage.

The oxidation stability of goat milk powder is affected by many factors, including the characteristic of the raw milk, processing technologies and storage conditions (Li et al., 2019). Martysiak-Żurowska et al. (2017) reported that UV-C radiation could cause photooxidation in breast milk components, particularly effecting lipids, and promote free radical degradation of lipids and proteins in human milk. Bandla, Choudhary, Watson, and Haddock (2012) used a continuous flow coiled tube ultraviolet reactor to treat milk, and found that UV-C had a significant effect on fat oxidation. The presence of HMF is an important indicator of the extent of Maillard reaction in milk powder

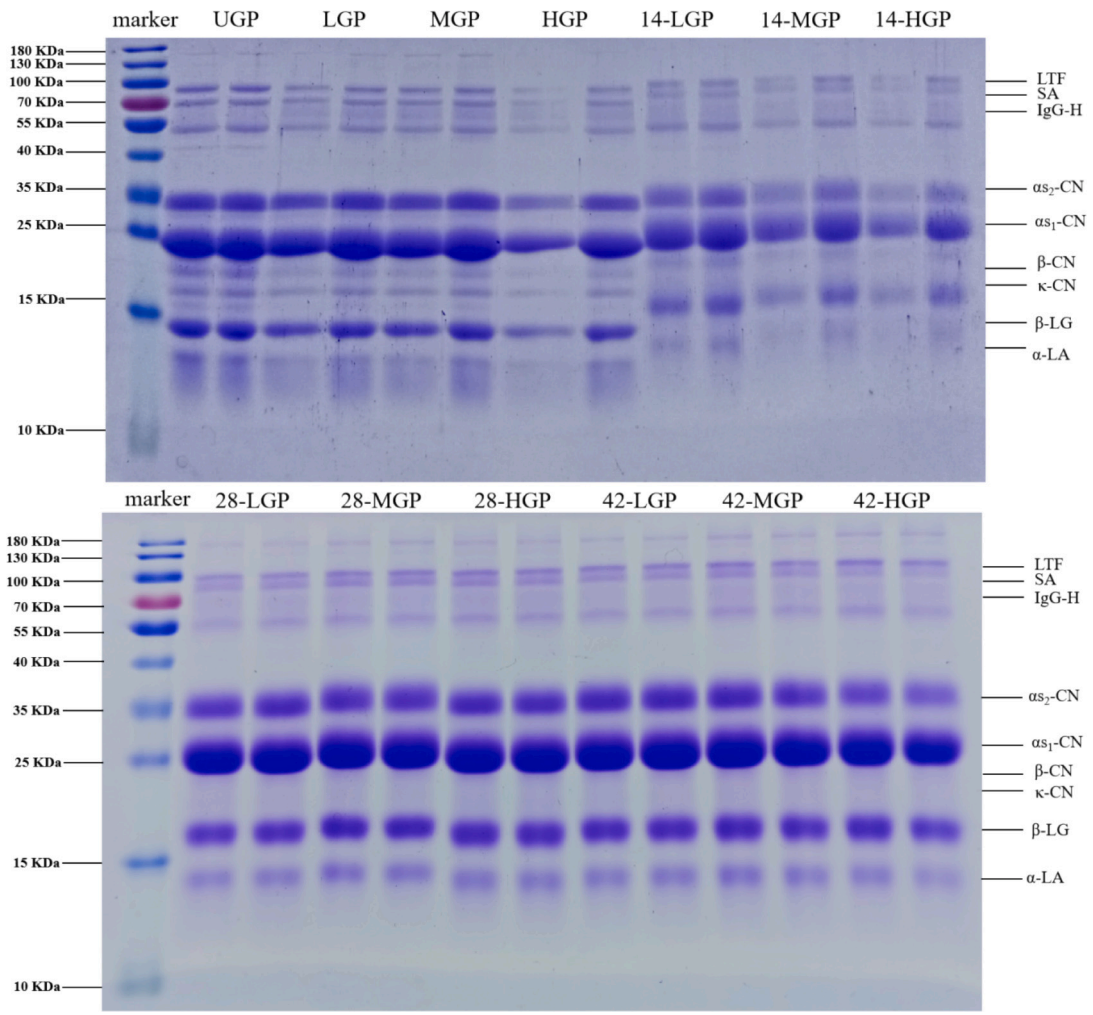

Fig. 3. Changes of protein components in UV-C radiation sterilized freeze-dried goat milk powder during accelerated storage test. UGP: unsterilized goat milk powder; LGP: low-dose UV-C radiation sterilized freeze-dried goat milk powder; MGP: medium-dose UV-C radiation sterilized freeze-dried goat milk powder; HGP: high-dose UV-C radiation sterilized freeze-dried goat milk powder. 14, 28 and 42 indicates storage days.

(Chávez-Servín, de la Torre Carbot, García-Gasca, Castellote, & López-Sabater, 2015). Peroxide, primarily resulting from lipid oxidation, are commonly measured via POV, which reflects the intial stages of oxidation in milk powder and indicates the presence of hydrogene peroxide. In the accelerated storage test, the percent of POV and HMF value of UV-C radiation sterilized freeze-dried goat milk powder to UGP was >1. With storage time, the percent showed an increasing trend (Fig. S1), indicating a gradual increase in POV and HMF content of goat milk powder, indicative of ongoing lipid oxidation and Maillard reactions. TBA reactive substances are a group of substances produced by the further degradation of lipid hydroperoxides, as measured by the TBA value, a primary parameter for further oxidation of milk fat. At 0 d, the percent of TBA value of goat milk powder to UGP was <1, but from 7 to 42 d, the ratio exceeded 1, and continued to increased as storage time progressed. The rising levels of TBA, POV and HMF significantly had an impact on the sensory quality of milk powder (Scheidegger et al., 2013).

In this study, the a* value of LGP was significantly positively correlated with HMF ($P < 0.05$). Additionally, the b* and ΔE values were very significantly positively correlation with HMF ($P < 0.01$). For the MGP, the a*, b* and ΔE values were very significantly positively correlated with HMF ($P < 0.01$). In HGP, the a*, b* and ΔE values also displayed significantly positive correlation with HMF ($P < 0.05$), as indicated in Table S1-S3. These findings suggested that the color changes observed in milk powder were primarilly due to the formation of yellow-brown macromolecular substances generated by the Maillard reaction during storage. Similarly, Thomsen, Lauridsen, Skibsted, and Risbo (2005) also tracked the progress of the Maillard reaction in whole milk powder by measuring HMF, color, and furosine levels in the early reaction products.

4. Conclusions

We investigated the effects of varying doses of UV-C radiation sterilization on the quality properties and storage stability of freeze-dried goat milk powder. After sterilization, the levels of total bacteria count and coliform bacteria in the goat milk powder complied the Chinese milk powder standards, and no noticeable color changes were observed by the naked eyes. The low moisture content and Aw in the LGP, MGP, and HGP samples contributed to a homogeneous and stable emulsion upon reconstitiution. Throughout the 42 d accelerated storage test, the microbial quality of goat milk powder remined good. Over time, there was an increased in the a*, b*, ΔE values, and the content of POV, HMF and TBA. Furthermore, color changes were strongly correlated with HMF levels, and there was no significant loss of active proteins in LGP, MGP, and HGP during storage. Briefly, the UV-C radiation sterilized freeze-dried goat milk powder maintained a high quality, suggesting that that this full process non-thermal processing technology is promising for producing high-quality milk powder.

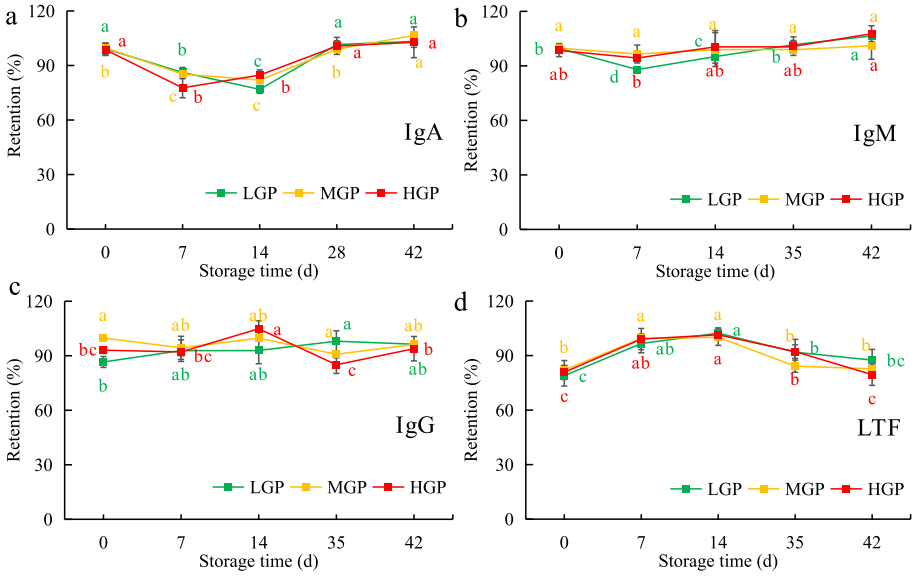

Fig. 4. Retentions of IgA (a), IgM (b), IgG (c), and lactoferrin (LTF) (d) in UV-C radiation sterilized freeze-dried goat milk powder during accelerated storage test. Different letters indicate a significant difference ($P < 0.05$) between the different storage times. LGP: low-dose UV-C radiation sterilized freeze-dried goat milk powder; MGP: medium-dose UV-C radiation sterilized freeze-dried goat milk powder; HGP: high-dose UV-C radiation sterilized freeze-dried goat milk powder. a-c: Different letters indicate a significant difference ($P < 0.05$) between the different storage times.

CRediT authorship contribution statement

Zhezhe Yu: Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Data curation. **Shangchen Fu:** Visualization, Validation, Resources, Methodology, Formal analysis, Data curation. **Linqiang Li:** Writing – original draft, Supervision, Resources, Investigation, Formal analysis. **Yongfeng Liu:** Visualization, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

None.

Data availability

Data will be made available on request.

Acknowledgments

The authors acknowledge the financial support from Shaanxi science and technology plan projects of China (2022KXJ-010, 2022ZDLNY04-09), Xi'an city science and technology plan projects of China (22NYGG0012, 23KGDW0021-2022), Science and technology plan projects in Xianyang city of Shaanxi Province (2021ZDZX-NY-0014).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101495.

References

Ansari, J. A., Ismail, M., & Farid, M. (2019). Investigate the efficacy of UV pretreatment on thermal inactivation of *Bacillus subtilis* spores in different types of milk. *Innovative Food Science & Emerging Technologies, 52*, 387–393. https://doi.org/10.1016/j.ifset.2019.02.002

Bandla, S., Choudhary, R., Watson, D. G., & Haddock, J. (2012). Impact of UV-C processing of raw cow milk treated in a continuous flow coiled tube ultraviolet reactor. *Agricultural Engineering International: CIGR Journal, 14*(2), 86–93.

Celestino, E. L., Iyer, M., & Roginski, H. (1997). The effects of refrigerated storage of raw milk on the quality of whole milk powder stored for different periods. *International Dairy Journal, 7*(2), 119–127. https://doi.org/10.1016/S0958-6946(96)00041-6

Chávez-Servín, J. L., Castellote, A. I., & López-Sabater, M. C. (2006). Evolution of potential and free furfural compounds in milk-based infant formula during storage. *Food Research International, 39*(5), 536–543. https://doi.org/10.1016/j.foodres.2005.10.012

Chávez-Servín, J. L., de la Torre Carbot, K., García-Gasca, T., Castellote, A. I., & López-Sabater, M. C. (2015). Content and evolution of potential furfural compounds in commercial milk-based infant formula powder after opening the packet. *Food Chemistry, 166*, 486–491. https://doi.org/10.1016/j.foodchem.2014.06.050

Christen, L., Lai, C. T., Hartmann, B., Hartmann, P. E., & Geddes, D. T. (2013). Ultraviolet-C irradiation: A novel pasteurization method for donor human milk. *PLoS One, 8*(6), Article e68120. https://doi.org/10.1371/journal.pone.0068120

Cilliers, F. P., Gouws, P. A., Koutchma, T., Engelbrecht, Y., Adriaanse, C., & Swart, P. (2014). A microbiological, biochemical and sensory characterisation of bovine milk treated by heat and ultraviolet (UV) light for manufacturing Cheddar cheese. *Innovative Food Science & Emerging Technologies, 23*, 94–106. https://doi.org/10.1016/j.ifset.2014.03.005

Fernandez, M., Ganan, M., Guerra, C., & Hierro, E. (2014). Protein oxidation in processed cheese slices treated with pulsed light technology. *Food Chemistry, 159*, 388–390. https://doi.org/10.1016/j.foodchem.2014.02.165

Gayán, E., Condón, S., & Álvarez, I. (2014). Biological aspects in food preservation by ultraviolet light: A review. *Food and Bioprocess Technology, 7*(1), 1–20. https://doi.org/10.1007/s11947-013-1168-7

GB 19644-2010. (2010). *National Standard of the People's Republic of China: national food safety standard: milk powder*.

GB 4789.2-2016. (2016). *National Standard of the People's Republic of China: national food safety standard food microbiological examination: aerobic plate count*.

GB 4789.3-2016. (2016). *National Standard of the People's Republic of China: national food safety standard food microbiological examination: coliforms*.

GB 5009.3-2016. (2016). *National Standard of the People's Republic of China: national food safety standard: moisture content*.

Guerrero-Beltr n, J. A., & Barbosa-C novas, G. V. (2004). Advantages and limitations on processing foods by UV light. *Food Science and Technology International, 10*(3), 137–147. https://doi.org/10.1177/1082013204044359

Harizi, N., Madureira, J., Zouari, A., Ayadi, M. A., Cabo Verde, S., & Boudhrioua, N. (2023). Effects of spray drying, freeze drying and gamma irradiation on the antioxidant activities of camel and cow milk fractions. *Processes, 11*(3), 897. https://doi.org/10.3390/pr11030897

Hassan, A. B., Al Maiman, S. A., Sir Elkhatim, K. A., Elbadr, N. A., Alsulaim, S., Osman, M. A., & Mohamed Ahmed, I. A. (2020). Effect of UV-C radiation treatment on microbial load and antioxidant capacity in hot pepper, fennel and coriander. *LWT - Food Science and Technology, 134*, Article 109946. https://doi.org/10.1016/j.lwt.2020.109946

Hu, G., Zheng, Y., Wang, D., Zha, B., Liu, Z., & Deng, Y. (2016). Comparison of microbiological loads and physicochemical properties of raw milk treated with single−/multiple-cycle high hydrostatic pressure and ultraviolet-C light. *High Pressure Research, 36*(4), 610. https://doi.org/10.1080/08957959.2015.1063626

Husu, J., Syväoja, E. L., Ahola-Luttila, H., Kalsta, H., Sivelä, S., & Kosunen, T. U. (1993). Production of hyperimmune bovine colostrum against *campylobacter jejuni. The Journal of Applied Bacteriology, 74*(5), 564–569.

Ji, X., Li, X., Ma, Y., & Li, D. (2017). Differences in proteomic profiles of milk fat globule membrane in yak and cow milk. *Food Chemistry, 221*, 1822–1827. https://doi.org/10.1016/j.foodchem.2016.10.097

Keklik, N. M., Elik, A., Salgin, U., Demirci, A., & Koçer, G. (2019). Inactivation of *Staphylococcus aureus* and *Escherichia coli* O157:H7 on fresh kashar cheese with pulsed ultraviolet light. *Food Science and Technology International, 25*(8), 680–691. https://doi.org/10.1177/1082013219860925

Li, S., & Shah, N. P. (2015). Effects of *Pleurotus eryngii* polysaccharides on bacterial growth, texture properties, proteolytic capacity, and angiotensin-I-converting enzyme-inhibitory activities of fermented milk. *Journal of Dairy Science, 98*(5), 2949–2961. https://doi.org/10.3168/jds.2014-9116

Li, Y. H., Wang, W. J., Guo, L., Shao, Z. P., & Xu, X. J. (2019). Comparative study on the characteristics and oxidation stability of commercial milk powder during storage. *Journal of Dairy Science, 102*(10), 8785–8797. https://doi.org/10.3168/jds.2018-16089

Liu, Y., Xiong, L., Kontopodi, E., Boeren, S., Zhang, L., Zhou, P., & Hettinga, K. (2020). Changes in the milk serum proteome after thermal and non-thermal treatment. *Innovative Food Science & Emerging Technologies, 66*, Article 102544. https://doi.org/10.1016/j.ifset.2020.102544

Martysiak-Żurowska, D., Puta, M., Kotarska, J., Cybula, K., Malinowska-Pańczyk, E., & Kołodziejska, I. (2017). The effect of UV-C irradiation on lipids and selected biologically active compounds in human milk. *International Dairy Journal, 66*, 42–48. https://doi.org/10.1016/j.idairyj.2016.10.009

Matak, K. E., Churey, J. J., Worobo, R. W., Sumner, S. S., Hovingh, E., Hackney, C. R., & Pierson, M. D. (2005). Efficacy of UV light for the reduction of *listeria monocytogenes* in goat's milk. *Journal of Food Protection, 68*(10), 2212–2216. https://doi.org/10.4315/0362-028X-68.10.2212

Matak, K. E., Sumner, S. S., Duncan, S. E., Hovingh, E., Worobo, R. W., & Hackney, C. R. (2007). Effects of ultraviolet irradiation on chemical and sensory properties of goat milk. *Journal of Dairy Science, 90*(7), 3178–3186. https://doi.org/10.3168/jds.2006-642

Ramos, G. L. P. A., Esper, L. M. R., & Gonzalez, A. G. M. (2023). A review on the application of UV-C treatment on food and food surfaces: Association with food microbiology, predictive microbiology and quantitative microbial risk assessment. *International Journal of Food Science and Technology, 59*(3), 1187–1196. https://doi.org/10.1111/ijfs.16880

Reddy, R. S., Ramachandra, C. T., Hiregoudar, S., Nidoni, U., Ram, J., & Kammar, M. (2014). Influence of processing conditions on functional and reconstitution properties of milk powder made from Osmanabadi goat milk by spray drying. *Small Ruminant Research, 119*(1), 130–137. https://doi.org/10.1016/j.smallrumres.2014.01.013

Salcedo, I., Andrade, J. A., Quiroga, J. M., & Nebot, E. (2007). Photoreactivation and dark repair in UV-treated microorganisms: Effect of temperature. *Applied and Environmental Microbiology, 73*(5), 1594–1600. https://doi.org/10.1128/AEM.02145-06

Sanchez, L. J., Zhu, D., Frew, R., & Kebede, B. (2020). Optimization of nuclear magnetic resonance and gas chromatography-mass spectrometry-based fingerprinting methods to characterize goat milk powder. *Journal of Dairy Science, 104*(1), 102–111. https://doi.org/10.3168/jds.2020-18467

Scheidegger, D., Radici, P. M., Vergara-Roig, V. A., Bosio, N. S., Pesce, S. F., Pecora, R. P., … Kivatinitz, S. C. (2013). Evaluation of milk powder quality by protein oxidative modifications. *Journal of Dairy Science, 96*(6), 3414–3423. https://doi.org/10.3168/jds.2012-5774

Stevenson, A., Cray, J. A., Williams, J. P., Santos, R., Sahay, R., Neuenkirchen, N., … Hallsworth, J. E. (2015). Is there a common water-activity limit for the three domains of life? *The ISME Journal, 9*(6), 1333–1351. https://doi.org/10.1038/ismej.2014.219

Sun, T. T. (2013). *Influence of common technological treatment on richfat-milk oxidation stability*. Master's thesis. Harbin, China: School of Food Science and Engineering, Harbin Institute of Technology.

Tedeschi, T., Aspri, M., Loffi, C., Dellafiora, L., Galaverna, G., & Papademas, P. (2023). Processing of raw donkey milk by pasteurisation and UV-C to produce freeze-dried milk powders: The effect on protein quality, digestibility and bioactive properties. *LWT - Food Science and Technology, 173*, Article 114404. https://doi.org/10.1016/j.lwt.2022.114404

Thomsen, M. K., Lauridsen, L., Skibsted, L. H., & Risbo, J. (2005). Temperature effect on lactose crystallization, Maillard reactions, and lipid oxidation in whole milk powder. *Journal of Agricultural and Food Chemistry, 53*(18), 7082–7090. https://doi.org/10.1021/jf050862p

Tribst, A. A. L., Falcade, L. T. P., Ribeiro, L. R., Júnior, B. R. D. C. L., & Oliveira, M. M. D. (2019). Impact of extended refrigerated storage and freezing/thawing storage combination on physicochemical and microstructural characteristics of raw whole and skimmed sheep milk. *International Dairy Journal, 94*, 29–37. https://doi.org/10.1016/j.idairyj.2019.02.013

Yu, H., Zheng, Y., & Li, Y. (2015). Shelf life and storage stability of spray-dried bovine colostrum powders under different storage conditions. *Journal of Food Science and Technology, 52*(2), 944–951. https://doi.org/10.1007/s13197-013-1046-3

Yu, Z., Qiao, C., Zhang, X., Yan, L., Li, L., & Liu, Y. (2021). Screening of frozen-thawed conditions for keeping nutritive compositions and physicochemical characteristics of goat milk. *Journal of Dairy Science, 104*(4), 4108–4118. https://doi.org/10.3168/jds.2020-19238

Zhang, W., Liu, Y., Li, Z., Xu, S., Hettinga, K., & Zhou, P. (2021). Retaining bioactive proteins and extending shelf life of skim milk by microfiltration combined with ultraviolet-C treatment. *LWT - Food Science and Technology, 141*, Article 110945. https://doi.org/10.1016/j.lwt.2021.110945