Manufacture of low lactose concentrated ultrafiltered-diafiltered retentate from buffalo milk and skim milk

Puneet Solanki · Vijay Kumar Gupta

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Abstract Lactose concentration was reduced by 68.64 and 74.64 % in buffalo milk and skim milk by their respective 3.05 fold and 4.4 fold UF-DF concentration. The maximum UF-DF concentration of buffalo milk to 66.65 % volume reduction was observed as compared to 74.35 % volume reduction in buffalo skim milk. Average initial permeate flux rate of buffalo milk (42.86 l/h/m^2) was much lower than skim milk (71.43 l/h/m^2), which dropped to 2.86 and 5.95 l/h/m^2 during UF-DF concentration. The initial permeate flux rate of homogenized buffalo milk (26.79 l/h/m^2) was comparatively lower than that of buffalo milk which dropped to 2.38 l/h/m^2 after 66.33 % volume reduction and 3.02 fold UF-DF concentration.

Keywords Lactose · Lactose reduction · Buffalo milk · Buffalo skim milk · Ultrafiltration · Diafiltration · Ultrafiltered-diafiltered retentate

Ultrafiltration (UF) is the most preferred method employed to extract milk protein used in the manufacture of milk protein concentrate in the dairy industry. It is also the most commonly used physical filtration process to remove lactose from milk. UF is an attractive process for reducing lactose from dairy products because it has molecular weight cut-off (MWCO) in the range of 1,000–5,00,000 Dalton. Therefore, lactose can

easily pass through the membrane while retaining all the protein in the retentate. Limsawat and Pruksasri (2010) who studied the effect of process parameters transmembrane pressure and feed flow rate on permeate flux and rejections of protein and lactose in commercial low-fat UHT milk concluded that a high degree of removal of lactose from milk could be achieved by UF with a minimal or no loss of protein in permeate. Ultrafiltration membranes reject almost about 100 % lipids and proteins and all substances associated with them. Hence, their concentration in the retentate increases in proportion to the concentration factor. Since, the rejection of lactose by ultrafiltration membrane is virtually zero, almost all of it passes into permeate. Dushkova and Dinkov (2005) investigating the dry matter, protein and fat concentration factor and protein retention coefficient in retentates obtained by ultrafiltration of cow skim milk and cow milk standardized revealed that almost all protein substances casein and whey proteins are retained on the membranes, whereas only non-protein soluble low molecular weight nitric compounds pass into the permeate. It is also evident that for standardized milk at the same volume reduction factors the fat concentration factor is approximately equal to the protein concentration factor. Patel and Mistry (1997) also reported that the rejection coefficients after 77 % weight reduction of UF buffalo skim milk were about 100 % for fat, ~ 95 % for proteins and over 50 % for ash. The incomplete rejection of proteins might have been due in part to the distribution of pore sizes in the membrane and in part to the distribution of molecular mass among the milk proteins. Additionally, the method used for determining protein quantitated non-protein nitrogen (NPN) as well as true protein. Most of the NPN compounds of milk were small enough to permeate the membrane during UF. Over 50 % of the ash components were rejected by the membrane because most of the soluble minerals were able to permeate the membrane, and only those minerals that were in colloidal state and associated with proteins were retained by the membrane. Rejection rates for all the components were

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P. Solanki (✉)
Food Technology Department, Maharshi Dayanand University,
Rohtak 124001, India
e-mail: puneetsol@gmail.com

V. K. Gupta
Dairy Technology Division, National Dairy Research Institute,
Karnal 132001, India

typical of those found for cow milk. Out of the total milk produced in India 132 million tones about 50–55 % is buffalo milk (IDF 2012). The total buffalo milk production in world is estimated around 97 million tones, which is 13 % of the total milk production in the world i.e. 749 million tones (IDF 2012). Buffalo milk is preferred for the manufacture of a large number of dairy products because of high total solids and fat content. Research workers have made attempts to use buffalo milk for the manufacture of evaporated milk, sweetened condensed milk, milk powders, infant milk foods and other dehydrated and formulated products. The low solubility of lactose has always been a problem in concentrated or condensed milk, as it could cause sandiness due to improper processing and storage. The buffalo milk and skim milk ultrafiltered-diafiltered (UF-DF) retentate with reduced lactose content presents itself as a good base for the preparation of concentrated or condensed milk and other formulated products. The aim of this work was to study ultrafiltration behaviour of buffalo milk and skim milk in respect of permeate flux rate variation and lactose reduction at different volume concentrations to manufacture low lactose concentrated UF-DF retentate.

Materials and methods

The raw buffalo milk obtained from the herd of Murrah buffalo and pasteurized buffalo skim milk was collected from the Experimental Dairy of the National Dairy Research Institute (NDRI), Karnal, India. The raw buffalo milk (90 Kg) or pasteurized buffalo skim milk (200 Kg) was stabilized by adding monosodium phosphate followed by forewarming at 90 °C for 10 min, filtered through a muslin cloth and then ultrafiltered at 50 °C by using a pilot ultrafiltration plant (Tech-Sep, France) with tubular module (channel diameter, 6 mm) having ZrO₂ membrane (membrane surface area 1.68 m² and membrane molecular weight cut off 50,000 Da). For trials on homogenized buffalo milk, the stabilized buffalo milk (forewarmed at 90 °C/10 min) was cooled to 70 °C and two stage-homogenized at 2,000 psi/500 psi and then subjected to ultrafiltration at 50 °C.

The quantity of permeate was collected at 10 min intervals and measured using measuring cylinder for permeate flux estimation in UF as well as Diafiltration (DF). Permeate flux rate was determined by measuring the amount of permeate coming out of the membrane in a given time and in a given area of the membrane and it was expressed in terms of l/h/m².

Volume reduction was calculated by using the formula: $(VR = VP/VO \times 100)$, where, VR = % Volume reduction, VP = Volume of permeate removed (Litres), VO = Original volume of milk (Litres). When ~ 2.5 fold and 4 fold volume concentration ratio of buffalo milk and skim milk, respectively were reached the DF was done. The hot water at 50 °C in

equal quantity to the retentate was added and the process continued till the final concentration level was achieved.

Sampling The 500 ml sample of buffalo milk and skim milk its UF and UF-DF retentate at different total solids concentrations was collected for the analysis. These samples were stored in the refrigerator (5±1 °C) till the analysis was carried out. The buffalo milk and skim milk final UF-DF retentate as obtained was hygienically collected in the pre-washed and steamed 20/40 l milk can and shifted to cold store room (1.0±1 °C).

Analysis The pH meter (Phan Labindia, Labtek Engg Pvt Ltd, India) was used to determine the pH of buffalo milk and skim milk and its UF-DF retentate at 20 °C. The total solids content of buffalo milk and skim milk its UF and UF-DF retentate was determined gravimetrically (BIS 2001) as for milk. The fat in Buffalo milk was determined by Gerber method as recommended by BIS (2001). The fat content in buffalo milk and skim milk its UF and UF-DF retentate was determined by weighing 15–20 g of sample in a beaker. This was transferred quantitatively into volumetric flask and the volume was made up to 100 ml. The 10.75 ml of diluted sample was measured into butyrometer of 10 % scale containing 10 ml of Gerber acid (BIS 2001). Total protein (Nx6.38) was estimated by semi micro-kjeldhal method as suggested by Meneffee and Overman (1940). Lactose was determined by the phenol-sulphuric acid method as described by Lawrence (1968) and ash was determined gravimetrically as per the method of BIS (2001) for milk with slight modifications.

Statistical analysis The experiments were carried out in triplicates. The data obtained from various experiments were subjected to analysis of variance as described by Snedecor & Cochran (1994) using Analysis ToolPak of Microsoft Excel.

Results and discussion

Preliminary studies on UF-DF concentration of buffalo milk and skim milk and improvement in its heat stability The pasteurized buffalo skim milk of average 10.12 % total solids was ultrafiltered up to 30.54 % total solids followed by diafiltration up to final concentration of 29.16 % total solids, to reduce maximum possible lactose. The buffalo skim milk UF-DF retentate thus obtained was pasteurized at 85 °C and cooled to room temperature (30 °C). Both, buffalo skim milk UF retentate and pasteurized UF-DF retentate were stored over night in refrigerator (5±1 °C) and in the cold store (1.0 ±1 °C). However, both the UF retentate and UF-DF retentate were destabilized next morning. As also reported by Patel et al. (1999) with the increase in the total solids during UF concentration of buffalo skim milk, the heat stability got

decreased. They reported that the initial heat coagulation time (HCT) at 120 °C of buffalo skim milk was 111 min at 10.59 % total solids (4.19 % protein) that decreased to 11 min after UF concentration to 28.39 % total solids (21.45 % protein). Therefore, the improvement in heat stability of buffalo skim milk was carried out by addition of stabilizing salts such as trisodium citrate, monosodium phosphate and disodium phosphate in order to stabilize its resultant UF-DF retentate. The addition of monosodium phosphate at 0.15 to 0.2 % levels to buffalo skim milk followed by forewarming at 90 °C/10 min, improved HCT at 140 °C from 8 min to 46–49 min (Solanki and Gupta 2009). The stabilized buffalo skim milk was then subjected to UF-DF concentration to prepare low lactose UF-DF retentate.

In case for the preparation of UF-DF buffalo milk retentate initial pH 6.78–6.71 of buffalo milk were observed to decrease to pH 6.48–6.43 after the addition of monosodium phosphate at 0.15 and 0.2 % levels followed by forewarming at 90 °C/10 min. The retentate prepared by UF-DF concentration of this buffalo milk was used for the preparation of low lactose concentrated milk but it destabilized on addition of sugar and heating to 70 °C. Therefore, instead of adding the 0.15 and 0.2 % monosodium phosphate to buffalo milk, its levels were increased to 0.25 to 0.3 %, in order to adjust the pH similar to that shown by stabilized buffalo skim milk (pH 6.35–6.30). The addition of monosodium phosphate at these levels followed by forewarming at 90 °C/10 min had the same effect on pH and heat stability of buffalo milk as observed in the buffalo skim milk. So, for all further studies the stabilization of buffalo milk was done through adjustment of its pH to 6.30–6.35 by addition of monosodium phosphate followed by forewarming at 90 °C/10 min.

Permeate flux rate of stabilized buffalo milk and skim milk and homogenized buffalo milk during UF-DF concentration The volume reduction in buffalo milk and skim milk by UF-DF concentration causes the increase of total solids especially protein which exerts great control over permeate flux. It is clear from Fig. 1 that the permeate flux rate decreases with percent volume reduction in buffalo milk and skim milk through UF-DF concentration. The initial permeate flux rate of stabilized buffalo skim milk was 71.43 l/h/m² which dropped to 9.82 l/h/m² at 4 fold UF concentration and further dropped down to 5.95 l/h/m² at 4.4 fold UF-DF concentration. While, the initial permeate flux rate of stabilized buffalo milk was 42.86 l/h/m² which dropped to 7.14 l/h/m² at 2.75 fold UF concentration which further dropped down to 2.86 l/h/m² after diafiltration to 3.05 fold UF-DF concentration. The maximum UF-DF concentration of buffalo milk to 66.65 % volume reduction was observed as compared to 74.35 % volume reduction in buffalo skim milk. There was no further reduction in volume of buffalo milk and skim milk because permeate flux rate became extremely low. On the other hand, the initial

Fig. 1 Change in permeate flux rate during ultrafiltration of buffalo milk and skim milk

permeate flux rate of homogenized buffalo milk was comparatively low being more viscous 26.79 l/h/m² and it dropped down to 5.95 l/h/m² and 2.38 l/h/m² at 2.85 fold UF and 3.02 fold UF-DF concentration, respectively. Kielczewska et al. (2003) who studied the effect of homogenization pressure on some physico-chemical properties of milk concluded that high-pressure homogenization induces a slight increase in viscosity and a decrease in heat stability of milk. The homogenized milk viscosity was always slightly higher than that of the control milk sample. The effect of homogenization on the change of the milk colloidal stability and viscosity results from an increase in milk fat dispersion degree and the subsequent adsorption of milk proteins on the inter-phase surface. It was found that in all the cases permeate flux rate drop suddenly after ~50 % volume reduction and so the efficiency of ultrafiltration got decreased. The initial permeate flux rate during UF-DF concentration of homogenized buffalo milk (26.79 l/h/m²) was comparatively much lower than that of buffalo milk (42.86 l/h/m²). Even then, the maximum possible UF-DF concentration of homogenized buffalo milk up to 66.33 % volume reduction could be achieved similar to that observed for buffalo milk. According to Patel et al. (1992) and Patel and Reuter (1985) on ultrafiltration of buffalo skim milk, the permeate flux declined from the very beginning falling rapidly initially. The flux then eventually levelled off at a rate, which was dependent on the degree of concentration and the flow conditions prevailing near the membrane surface. They also reported that much lower permeate flux rate in buffalo milk compared to buffalo skim milk was due to the higher total solids and its fat content, which played a considerable role in deposit formation on the UF membranes.

Table 1 Composition of buffalo milk and skim milk, its UF and UF-DF retentate and lactose reduction, on dry matter basis

	Buffalo skim milk ^a	UF retent. ^a	UF-DF retent. ^a	Buffalo milk ^b	UF retent. ^b	UF-DF retent. ^b
Total solids (%)	10.12±0.21	20.53±1.76	23.02±0.97	16.89±0.43	38.08±1.06	38.32±0.75
Fat (%)	0.10±0.05	0.23±0.06	0.37±0.06	6.90±0.44	19.60±1.18	20.41±0.72
Protein (%)	4.25±0.07	14.02±1.77	17.65±0.88	4.28±0.05	12.11±0.29	12.68±0.60
Lactose (%)	5.01±0.06	4.43±0.17	2.89±0.05	4.94±0.05	4.60±0.04	3.51±0.06
Ash (%)	0.77±0.03	1.84±0.10	2.12±0.09	0.78±0.02	1.76±0.07	1.71±0.06
Lactose, on dry matter basis, (%)	49.50±0.42	21.52±2.47	12.56±0.73	29.24±0.51	12.09±0.39	9.17±0.06
Lactose reduction, on dry matter basis, (%)	–	56.55±4.62	74.64±1.26	–	58.64±0.95	68.64±0.41

^a mean from 3 trials^b mean from 5 trials

Effect of UF and UF-DF concentration on composition and lactose reduction of buffalo milk and skim milk and their retentates Average chemical compositions of buffalo skim milk, its UF and UF-DF retentates are shown in Table 1. Lactose concentration of buffalo skim milk was reduced from 5.01 to 2.89 % for a corresponding increase in total solids from 10.12 to 23.02 % as the protein content got increased from 4.25 to 17.65 % by UF-DF concentration. The lactose, on dry matter basis, was observed to reduce from 49.50 % in buffalo skim milk to 12.56 % in the UF-DF retentate. This represented a 74.64 % reduction of lactose in the buffalo skim milk at maximum possible UF-DF concentration. Patel (1996) reported that after 5.5 fold UF concentration of buffalo skim milk the lactose decreased from 5.1 % to 4.21 % with increase in total solids from 9.31 % to 24.52 %, protein from 3.32 % to 17.99 % and ash from 0.86 % to 2.33 %. While, the UF-DF retentate had 22.76 % total solids, 18.01 % protein, 2.55 % lactose and 2.19 % ash. Mistry (2002) and Mistry and Hassan (1991) also reported reduction in lactose from 4.91 to 3.81 % for a corresponding increase in total solids from 8.61 to 20.90 % by UF concentration of cow skim milk. This represented ~68 % reduction in lactose through UF up to 5:1 volumetric concentration ratio. During diafiltration three times with water to 21.57 % total solids, lactose concentration was further reduced to 0.08 %, which represented 99.7 % reduction in lactose. Vyas and Tong (2003) reported that about 76 % of the total lactose present permeated through the membrane when the cow skim milk was UF concentrated to 4 fold level.

Average chemical composition of buffalo milk, its UF and UF-DF retentates prepared with maximum possible UF-DF concentration is shown in Table 1. Lactose concentration of buffalo milk was reduced from 4.94 to 4.60 and 3.51 % for a corresponding increase in total solids from 16.89 to 38.08 and 38.32 % by its UF and UF-DF concentration. The protein content got increased from 4.28 to 12.11 and 12.68 % and fat from 6.90 to 19.60 and 20.41 %, respectively. The lactose, on dry matter basis, was observed to reduce from 29.24 % in buffalo milk to 12.09 and 9.17 % in its UF and UF-DF retentate. This represented a 58.64 and 68.64 % reduction in

lactose concentration of buffalo milk by its approximately 2.75 fold and 3.05 fold UF and UF-DF concentration. Lonergan et al. (1981) reported that about 68–72 % lactose reduction of cow whole milk could be done, by its ultrafiltration to approximately 3 folds UF concentration. Patel et al. (1992) reported that on ultrafiltration, the fat and total solids content got increased from initial 6 % fat and 15.73 % TS of buffalo milk to finally 19.40 % fat and 42.83 % TS in its UF retentate.

Conclusion

Lactose reduction of 74.64 % was achieved by approximately 74.35 % volume reduction in buffalo skim milk through its UF-DF concentration. However, the buffalo milk UF-DF retentate had only 68.64 % lactose reduction after 66.33 % volume reduction which was comparatively lower than buffalo skim milk. Their was difference in initial permeate flux rate of buffalo milk (42.86 l/h/m²) compared to buffalo skim milk (71.43 l/h/m²) due to higher total solids and presence of fat which enhances the deposits formation by whole milk as compared to skim milk. The initial permeate flux rate of homogenized buffalo milk (26.79 l/h/m²) was comparatively much lower for being more viscous than that of buffalo milk.

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