Characterisation of flux and composition during ultrafiltration and diafiltration of cheddar cheese whey

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Ultrafiltration (UF) and diafiltration (DF) of cheddar cheese whey have been carried out after adjusting pH of whey to various levels ranging from 3.0 to 7.2 and imparting different heat treatments in a pilot scale hollow fibre UF plant, possessing PM 50 polysulphone membrane. Concentration has been carried out at 50°C and 1.75 and 0.5 bar inlet and outlet pressures, respectively to a volume reduction of 95% followed by diafiltration in order to elicit their effect on flux and composition of resultant whey protein concentrate (WPC). It is evident from the results that a significantly higher flux could be obtained when whey was processed either at a pH of 3.0 or 7.2 along with a preheat treatment of 80°C/15 s compared to other levels of pH 4.5, 5.6 and 6.4 and heat treatment 60°C/30 min. By UF the protein content on dry matter increased to a level ranging from 61.48 to 63.82% from an initial value of 14.89%. The lactose content decreased to a level ranging from 25.60 to 28.50% from an initial value of 76.14%. Whereas the ash content to a level of 3.17 to 6.23% from 8.20%. The pH of processing had significant effect on ash content being minimum at pH 3.0 (3.17%) and maximum at pH 7.2 (6.30%). The calcium and the phosphorus content of retentate followed the similar pattern as that of ash.

Ultrafiltration (UF) is being widely explored in the dairy industry for recovery whey protein in the form of whey protein concentrate (WPC)1-2. Presently, it is possible to recover proteins from whey in their native form through UF process. Hence, the resultant WPC finds wide range of applications in various food formulations3-4. However, the flux during UF and composition of the resultant WPC are the prime factors for commercial exploitation of this technology for sustainable and economic operation5-6. The economy of the process is dependent on flux, whereas the end use of the resultant WPC is dependent on the composition of retentate7-8. Both flux and composition of WPC are dependent on composition, type, pH and ionic strength of whey9. Nevertheless, the flux can be altered by imparting selective treatment to whey prior to UF10. However, the kind of treatment to be induced vary widely with the type and composition of whey11. The flux and the composition of WPC can be monitored depending on the end use4-10. Whey obtained from buffalo milk cheddar cheese preparation vary widely with respect to pH and composition, compared to cow milk cheddar cheese whey12-13. The effect of various processing conditions on flux and composition of WPC during UF and diafiltration (DF) of buffalo milk cheddar cheese whey is yet to be made. It is necessary to monitor the flux for successful implementation of this technology in our country and the composition of WPC to study its use in various food applications. Hence, this investigation was undertaken to characterise the flux and composition during UF and DF of buffalo milk cheddar cheese whey.

Experimental Procedure

Buffalo milk cheddar cheese was prepared in National Dairy Research Institute, Karnal, following the method suggested by Kanawjia14 and the resultant whey was used in present investigation.

Ultrafiltration plant—A pilot scale hollow fibre UF plant possessing polysulphone membrane (Romicon, membrane type PM 50, i.e., 50000 mol. wt. cut off, effective area 1.4 m²) supplied by Alfa-Laval, Denmark was used in this investigation. The module consisted of cartridges containing 700 fibres oriented in parallel with a internal and external diameter of 0.51 and 1.1 mm respectively. The fibres are plotted in the resin at their ends enclosed in the permeate collecting tube. The feed was pumped from an external tank by the feed pump (at 1.75 bar pressure)
and fed into the circulation loop by using circulation pump (at 0.5 bar pressure) with a crossflow velocity of 2.5 m/s. Part of the liquid permeate through the fibre and is collected in the cartridges shell from where it leaves through the upper permeate outlet. The remaining liquid inside the fibres, the concentrate mixed with new feed and fed back to the circulation pump for another passage through cartridge.

Processing of whey—Clarified whey was adjusted to various pH levels, viz., 3.0, 4.5, 5.6, 6.4 and 7.2 either by addition of 10% sodium hydroxide or by 10% hydrochloric acid. At each trial 250 L fresh clarified pH adjusted buffalo milk cheddar cheese whey was subjected to either 60°C for 30 min or 80°C for 15 s heating and cooled to 50°C. UF was carried out separately in each combination at this temperature to a volume reduction of 95% by maintaining 1.75 and 0.50 bar inlet and outlet pressures, respectively.

Diafiltration—UF of 250 L of whey was carried to a level of 95% volume reduction with the above processing condition. When 95% volume reduction was attained, distilled water (50°C) was added to the retentate at 1:1 proportion. Filtration was carried out till all the added water was removed in the form of permeate. Similarly a second DF was carried out.

Volume reduction—This was estimated by using the following formula:

\[ \text{VR} = \frac{\text{VP}}{\text{VO}} \times 100 \]

Where, VR = percentage volume reduction, VP = volume of permeate removed (mL), and VO = initial volume of whey (mL).

Flux—At a regular interval of volume reduction the flux was determined by measuring the amount of permeate coming out of the membrane in a given time at the given area of membrane (1.4 m²) and expressed in terms of standard flux.

\[ J = \text{L m}^{-2} \text{ h}^{-1} \]

Degree of retention—At regular intervals of volume reduction the retentate was analysed for various compositional attributes and the components were expressed on dry matter basis. The yield of each component was calculated by the following formula.

\[ Y = \frac{C_1}{f} \cdot C_0 \]

Where, \( Y \) is the yield of component, \( C_0 \) is the initial concentration of the given component, \( C_1 \) is the final concentration of the given component, and \( f \) is the concentration factor.

Analytical methods—The total solids content was analysed by gravimetric method\(^1\). The fat content of the retentate was estimated by Gerber method using skim milk butyrometer\(^2\). The total nitrogen content was determined by a standard Kjeldahl method\(^3\). From the total nitrogen, the total protein was obtained by multiplying with a factor 6.38. Non-protein nitrogen (NPN) was estimated by determining the nitrogen content in the trichloracetic acid filtrate\(^4\) by Kjeldahl method. Lactose was estimated by the phenol sulphuric acid method\(^5\). The ash content was determined by incinerating the samples at 550°C in muffle furnace\(^6\), whereas calcium was assayed by the calmagite method\(^7\) and phosphorus by Fiske and Subbarao method\(^8\).

Results and Discussion

Flux during ultrafiltration—the effect of various pH adjustment and preheating of whey on the resultant UF flux is presented in Fig. 1. The clarified whey was subjected to heat treatments at various pH levels. The flux rate appeared to be better at extreme pH values. At pH 3.0 and 7.2, flux was significantly (\( P \leq 0.01 \)) higher than at other pH levels. The respective average flux at a pH of 3.0, 4.5, 5.6, 6.4 and 7.2 for a heat treatment of 60°C/30 min was 44.29, 20.88, 28.75, 34.36 and 46.88 L m\(^{-2}\) h\(^{-1}\). Similarly,
When whey was heated to 80°C/15 s, the flux rate was better compared to 60°C/30 min heating at all levels of pH. The mean flux values being 48.33, 22.77, 30.50, 36.29 and 51.71 L m⁻² h⁻¹ respectively for 95% volume reduction. It is evident from Figs 1a and 1b that as the volume reduction increased the flux decreased irrespective of the treatment imparted to the whey. However, the rate of decline in flux varied significantly (P ≤ 0.01) with the pH and the heat treatment of whey. This can be ascribed to the fact that UF performance is strongly dependent on physicochemical characteristics which refers to the solute interaction and solute membrane interaction²². The change in pH and heat treatment affects the status of calcium, salts and the structural configuration of protein and flux²²,²³.

When whey was processed at pH 7.2 or 3.0 the flux rate was observed to be higher than at native pH (6.4). At pH 4.5 the net charge on the proteins is low and hence, dispersion of protein is poor. They get adsorbed on the surface of the membrane, forming gel layer which results in lowest flux. As the pH was lowered from 4.5 the dispersion of proteins improves, calcium gets solubilized and pass through the membrane without much fouling²⁴.

At pH 5.6 lower flux was noted which could be attributed to the amorphous tricalcium phosphate and adsorption of beta-lactoglobulin as the pH is near to its isoelectric point⁹. The pronounced increase in the premeate flux at pH 7.2 may be due to heating of whey at elevated pH. Under such conditions a considerable precipitation of calcium phosphate is expected in such a form (probably as hydroxy apatite) that results in reduced fouling of membrane. Addition of calcium to whey and adjusting pH above 6.5 and heating increases the flux²⁵. As the calcium content of buffalo milk cheddar cheese whey is already higher heating at pH 7.2 probably resulted in calcium induced protein interaction which were found to be non-fouling. Heating whey to a temperature of 80°C/15 s resulted in better flux than at 60°C/30 min. Probably higher temperature is needed for apatite formation or calcium induced beta-lactoglobulin self aggregation and protein-protein interaction. Increase in flux by 50% was observed when cheese whey was heated to 80°C/15 s compared to pasteurization of whey¹⁰.

Effect of processing variables on the composition of WPC—The composition of retentate as affected by pH of whey and heat treatment is presented in Table 1. It is apparent from the results that the pH of whey has a significant (P ≤ 0.05) effect on the composition of the retentate. As could be seen from the table as the pH is increased from 3.0 to 7.2, the total ash, calcium and phosphorus content of the retentate increased at both the levels of heating. The yield of protein varied from 61.48 to 63.82%. The respective yield at pH 3.0, 4.5, 5.6, 6.4 and 7.2 was found to be 63.04, 61.80, 63.10, 63.79 and 62.40% for 80°C/15 s heating and it was 63.15, 61.48, 63.30, 63.82 and 62.80% for 60°C/30 min heating. The variation in protein yield when processed at different levels of pH could be attributed to variation in permeation behaviour of other constituents especially mineral matter rather than permeation behaviour of true proteins. Similar results are reported by other workers⁶,²⁶. Little variation in the NPN content of the retentate was observed with the change in pH of whey. The retentate had 1.02, 0.92, 1.03, 1.05 and 0.98% NPN at pH 3.0, 4.5, 5.6, 6.4 and 7.2, respectively when heated at 80°C/15 s as against 1.08, 0.96, 1.02, 1.06 and 0.98% at 60°C/30 min heating. The lowest yield of protein at pH 4.5 could attributed to lowest recovery of NPN rather than any of the true protein. The highest protein content at pH 6.4 was also due to high retention of NPN. The lactose content of retentate varied from 25.36 to 28.50%. The highest being at pH 3.0 and lowest at pH 7.2.

Wide variation in the ash content was observed when whey was ultrafiltered at different pH values and heat treatment. It is evident from table as the pH of UF increased from 3.0 to 7.2 the ash content of retentate increased dramatically at both the levels of heating. The lowest ash content was observed at pH 3.0 and the highest at pH 7.2. The ash content of the retentate at pH 7.2 was nearly double that of the retentate at pH 3.0. It was clear from the results that the ash content of the retentate could be monitored by

<table>
<thead>
<tr>
<th>Component</th>
<th>Heat treatment</th>
<th>pH of whey processing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protein</td>
<td>60°C</td>
<td>63.15 61.48 63.30 63.82 62.80</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>63.04 61.80 63.10 63.79 62.40</td>
</tr>
<tr>
<td>NPN</td>
<td>60°C</td>
<td>1.08 0.96 1.02 1.06 0.98</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>1.02 0.92 1.03 1.05 0.98</td>
</tr>
<tr>
<td>Lactose</td>
<td>60°C</td>
<td>28.30 26.50 27.15 27.42 25.60</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>28.50 26.40 27.05 27.32 25.36</td>
</tr>
<tr>
<td>Ash</td>
<td>60°C</td>
<td>3.27 3.78 4.02 4.28 6.30</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>3.17 3.98 4.03 4.25 6.23</td>
</tr>
<tr>
<td>Calcium</td>
<td>60°C</td>
<td>0.26 0.69 0.73 0.78 1.02</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>0.23 0.68 0.72 0.76 1.01</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>60°C</td>
<td>0.15 0.44 0.46 0.49 0.69</td>
</tr>
<tr>
<td></td>
<td>80°C</td>
<td>0.14 0.42 0.44 0.47 0.68</td>
</tr>
</tbody>
</table>

*Average of three trials
adjusting the pH of whey during ultrafiltration. The lowest ash content at pH 3.0 could be ascribed to the greater permeation of some minerals, especially calcium and phosphorus. These minerals become soluble from their colloidal state when the pH is lowered. The soluble minerals permeate easily through the membrane leading to lower ash content of the retentate. The results are in conformity with the earlier workers 8-24.

**Effect of diafiltration on the composition of retentate**—The effect of diafiltration of UF retentate on the changes in the composition characteristics as represented on dry matter basis is presented in Table 2. It is clear from the result that by ultrafiltration of whey to 95% volume reduction the protein content of retentate increased to as high as 86.20% protein by UF of whey followed by two stage DF. The increase in protein and decrease in lactose and ash content during UF and DF could be attributed to the addition of water to the UF retentate, which aids in reducing the viscosity and, thereby more of lactose, minerals and low molecular weight nitrogenous compounds pass along with permeate 28,29. During DF process it was observed that retention of calcium and phosphorus was high, whereas total ash retention decreased to a great extent. This could ascribed to the fact that repeated DF of whey results in removal of potassium, sodium and magnesium to a greater extent and calcium and phosphorus to a lesser extent 27. In the present study, higher retention of calcium and phosphorus was observed by the DF. Similar results showing higher retention of calcium and phosphorus during DF was reported by earlier workers 29,30.

### References

15 IS SP 18 (Part II) Indian Standards Institutions, Manak Bhavan, New Delhi, 1962.