



Effect of composition on moisture sorption of delactosed permeate

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ABSTRACT

Delactosed permeate (DLP) is an effluent generated after the separation of most of the proteins and lactose from cheese whey. The inability to obtain stable products with acceptable low moisture content is one of the major drawbacks to further commercial development of DLP. To determine the factors influencing the sorption behavior, DLP samples from three different dairy processing industries were characterized for their composition and sorption behavior. Sorption data were successfully fitted to the Peleg model. The relationships among the compositional elements and Peleg constants for initial sorption rate (K_1) and sorption capacity (K_2) showed total sugar, lactic acid and mineral content had a significant influence on sorption. With an increase in the lactic acid and mineral content, the K_1 and K_2 values decreased, indicating higher initial sorption rate and higher sorption capacity.

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1. Introduction

Delactosed permeate (DLP) is an effluent stream generated after the separation of most of the proteins and lactose from cheese whey. DLP is composed of milk minerals, acids, residual proteins, residual lactose and water. The concentrations of these solids in DLP depend on the various processes it undergoes and respective efficiencies of those processes. DLP is commonly utilized as animal feed with the rest being field spread (Vembu & Rathinam, 1997).

High water content of DLP (60–75%) makes it difficult to incorporate into food products without further modification. Although utilization of DLP in food products would enhance the profitability of the dairy industry, inability to obtain stable products with acceptable low moisture content is a major drawback to further commercial development. The reasons for stickiness and hygroscopicity of DLP are still unclear, although the presence of organic acids and minerals in DLP are thought to be responsible for both drying problems and post-drying instability. Study of water sorption behavior for DLP samples from different companies having varied composition should aid in understanding of stability-related problems of DLP.

To better understand the mode of water absorption by food materials, a two parameter non-exponential empirical model was proposed by Peleg (1988) (Eq. (1))

$$M = M_0 \pm \frac{t}{K_1 + K_2 t} \quad (1)$$

where, M is moisture content ($\text{g H}_2\text{O g}^{-1}$ dry matter (DM)) at time t (h), M_0 is initial moisture content ($\text{g H}_2\text{O g}^{-1}$ DM), K_1 is a Peleg rate

constant ($\text{h} \times (\text{g H}_2\text{O g}^{-1} \text{DM})^{-1}$) and K_2 is Peleg capacity constant ($(\text{g H}_2\text{O g}^{-1} \text{DM})^{-1}$). The Peleg rate constant K_1 , is related to the mass transfer rate, whereas K_2 is related to maximum water absorption capacity.

Lower K_1 and K_2 values imply higher initial sorption rate and sorption capacity, respectively. In Eq. (1), ‘±’ becomes ‘+’ if the process is sorption and ‘-’ if the process is desorption.

The applicability of Peleg’s model has been demonstrated for various food products like milk powder, rice, chickpea and grains (Peleg, 1988; Sopade, Ajisegiri, & Badau, 1992; Sopade, Xun, Halley, & Hardin, 2007; Turhan, Sayer, & Gunasekaran, 2002).

The present work was carried out to study the sorption characteristics of DLPs from different companies. This study would aid in identifying the primary constituents in DLP with the most influence on the characteristic sorption behavior of DLP, which in turn is responsible for stickiness and hygroscopicity.

2. Materials and methods

2.1. Materials

DLP samples labelled 1, 2 and 3 were procured from three local dairy processing industries. Sodium azide (0.03%, w/w) was added to each DLP sample to prevent microbial growth. The samples were stored at 7 °C until further use. All the chemicals and reagents used were of analytical grade and were purchased from Fisher Scientific, New Jersey, USA.

2.2. Compositional analysis

The moisture content of DLP samples was analyzed by a Karl Fischer (KF) titration system (Brinkmann Model 795, Metrohm USA

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Inc., Miami, FL, USA) and from that the total solid content was estimated using simple material balance principles. Protein content was determined using a modified Folin Lowry method (Bund & Pandit, 2007).

The sugar, acid and mineral profiles of DLP samples were analyzed by Covance Laboratories Inc. (Madison, WI, USA). The sugar profile was analyzed by gas chromatography using a flame ionization detector (Mason & Slover, 1971). Sugars were extracted from the sample with water. Aliquots were dried under inert gas and reconstituted with a hydroxylamine hydrochloride solution in pyridine containing phenyl- β -D-glucoside as the internal standard. The resulting oximes were converted to silyl derivatives with hexamethyldisilazane (HMDS) and trifluoroacetic acid (TFA) treatment and analyzed by GC. The organic acid profile was analyzed by HPLC and UV detection (AOAC, 2000). Samples were treated with acidic solution to extract organic acids. Samples were filtered, centrifuged and filtered again. Organic acids were separated then using HPLC. The mineral profile was analyzed by Inductively Coupled Plasma (ICP) spectrometry (AOAC, 2000). The samples were ashed at $500 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$ for 5–16 h. The resulting ash was treated with concentrated hydrochloric acid, dried and redissolved in hydrochloric acid solution. The amount of each element was determined using ICP spectrometry by comparing the emission of the unknown sample against the emission of each element from standard solution. Chloride content of DLP samples was analyzed as per the method described in official methods of analysis (AOAC, 2000). The weighed sample was added with silver nitrate (0.1 M) followed by halogen-free HNO_3 . The solution was boiled and KMnO_4 was added to the boiling solution. The solution was cooled, filtered and excess silver nitrate was titrated against 0.1 M KSCN using Fe alum as indicator.

2.3. Characterization of state of various forms of DLP samples

The crystalline state of both liquid DLP samples (as received) and the dried samples (dried over silica gel and used later for the moisture sorption kinetics) were characterized by using polarized light microscopy and X-ray powder diffraction (XRPD). A Nikon microscope (Labophot-2, Japan; Quicam camera, Fast 1394, Canada) with cross polarizers was used to indicate the presence or absence of crystalline material in the sample. Similarly, dried DLP samples were also analyzed using XRPD for further confirmation on the state of the material in the samples. XRPD was performed in the reflection mode with a Bruker D8 Advance X-ray diffractometer (Bruker AXS Inc., Madison, WI, USA), which was equipped with a Cu $K\alpha$ source ($\lambda = 1.54056 \text{ \AA}$) operating at a tube load of 40 kV and 40 mA. The divergence slit was 1 mm, the receiving slit was 1 mm, and the detector slit was 0.1 mm. Data were collected by a Sol-X detector. Each sample was scanned between 2 and 40° (2θ) with a step size of 0.02° and a maximum scan rate of 1 s step^{-1} .

2.4. Sorption isotherm study

Sorption experiments were performed under both equilibrium and dynamic conditions.

For equilibrium sorption isotherms, DLP samples of known weight were collected in water activity sample holders. The water from samples was either evaporated in a ventilation hood or extra water was added to reach the desired moisture content (\sim in the range of 0.05 – $19 \text{ g moisture g}^{-1}$ dry matter ($\text{g H}_2\text{O g}^{-1}$ DM)). Samples were then analyzed for water activity (a_w) using an Aqua lab water activity meter, model series – 3TE (Decagon Devices Inc., Washington, DC, USA) at constant temperature of $23 \text{ }^\circ\text{C}$ in continuous mode. Equilibrium was

established in about 1 h to several hours depending on moisture levels. The system was considered equilibrated when a difference in water activity value less than 0.001 in several sequential readings was observed. The moisture content was plotted against the respective water activity to generate equilibrium isotherms for each DLP sample.

For moisture sorption kinetic study (dynamic condition), about 5.5 g (exact weight recorded) of each DLP sample was collected in a water activity sample holder and first dried over silica gel. The resultant moisture content in the samples was in the range of 0.045 – $0.070 \text{ g H}_2\text{O g}^{-1}$ DM. The samples were then placed in desiccators over distilled water providing a relative humidity of 100% for moisture uptake. The desiccators were stored in a constant temperature ($23 \text{ }^\circ\text{C}$) environment. Samples were analyzed over time for water content by measuring the change in weight and for a_w using the water activity meter. The moisture content was calculated using principles of material balance with the weight gained at periodic interval of time and initial moisture content.

2.5. Application of Peleg model to sorption kinetics

The sorption kinetic behavior was analyzed with the Peleg model (Eq. (1)). The rate of sorption (R) can be obtained from the derivative of the Peleg equation. The Peleg rate constant K_1 relates to initial sorption rate (R_0) (i.e., R at $t = t_0$)

$$R_0 = \left. \frac{dM}{dt} \right|_{t_0} = \pm \frac{1}{K_1} \quad (2)$$

The Peleg capacity constant K_2 relates to maximum (or minimum) attainable moisture content. As $t \rightarrow \infty$, Eq. (1) gives the relation between equilibrium moisture content (M_e) and K_2

$$M|_{t^*} = M_e = M_0 \pm \frac{1}{K_2} \quad (3)$$

Dry basis moisture content ($\text{g H}_2\text{O g}^{-1}$ DM) was used for all calculations. The linearized form of Peleg equation (Eq. (4)) was used to regress the moisture content versus sorption time.

$$\frac{t}{M - M_0} = K_1 + K_2 t \quad (4)$$

The validation of fit of Peleg's model to the sorption data was done using SAS software (version 8, SAS Institute Inc., Cary, NC, USA). The P -values < 0.05 suggested a good fit of the model. Similarly, the relationships obtained for the sorption behavior in terms of Peleg's constants K_1 and K_2 with individual components of DLPs were validated using regression analysis in Excel, with significance level set at $\alpha \approx 0.1$.

3. Results and discussion

3.1. Composition of DLP samples

The characteristics and composition of the three DLP samples are shown in Table 1. The chemical composition of DLP depends on numerous factors including the composition of the original milk used for cheese making, the type of cheese and make procedures, holding time and temperature prior to whey pasteurization, and whey processing steps and conditions (i.e., whey protein separation, lactose refining, etc.). Even DLP from the same source is likely to vary on a regular basis. Thus, the composition of the three DLP samples used here provides only a snap shot of the range of DLP available. Still, these three samples exhibit a fairly wide variability in properties.

Table 1
Composition of delactosed permeate (DLP) samples procured from different dairy processing industries.

Test	DLP samples		
	1	2	3
Moisture content ^a	66.2	74.1	64.4
pH	5.6 ± 0.1	5.2 ± 0.1	5.2 ± 0.1
<i>Sugar profile^b</i>			
Fructose	<0.30	<0.39	<0.28
Galactose	2.66	<0.39	3.93
Glucose	1.18	<0.39	1.69
Sucrose	<0.30	<0.39	<0.28
Lactose	64.20	55.21	41.29
Maltose	<0.30	<0.39	<0.28
Total sugars	68.05	55.21	46.91
<i>Acid profile^b</i>			
Acetic acid	<0.15	<0.19	<0.14
Citric acid	5.74	3.46	4.92
Lactic acid	2.47	5.71	7.28
Malic acid	<0.15	<0.19	<0.14
Total acids	8.21	9.17	12.19
<i>Mineral profile^b</i>			
Calcium	0.86	0.69	1.12
Magnesium	0.21	0.23	0.30
Phosphorus	1.74	1.63	2.32
Potassium	3.93	6.87	8.37
Sodium	1.23	2.33	2.30
Chloride	1.31	5.25	5.45
Total minerals	9.29	17.00	19.86
Protein content ^b	1.36	2.14	2.37

^a Moisture content given as g H₂O 100 g⁻¹ DLP.

^b Sugars, acids, minerals and protein content given as g 100 g⁻¹ DM.

The moisture content of DLP samples was in the range of 64–75 g 100 g⁻¹ of DLP. The pH of DLP 1 was higher than that of DLP 2 and 3, which indicate lower acidity in DLP 1 compared with the others. As there was considerable variation in the moisture content of DLP samples, composition data with respect to sugar, acid and mineral profile were normalized to g 100 g⁻¹ DM.

The major sugar present in each DLP was lactose, which varied from a high of 64.2 g 100 g⁻¹ DM in DLP 1 to a low of 41.3 g 100 g⁻¹ DM in DLP 3. The amount of lactose present in DLP varies with the efficiency of removing lactose in the refining operation. The total sugar content was again highest in DLP 1 (68.1 g 100 g⁻¹ DM) and the lowest in DLP 3 (46.9 g 100 g⁻¹ DM). Glucose and galactose, by-products of microbial fermentation of lactose, were also found at low, but variable (0.4–3.9 g 100 g⁻¹ DM) levels.

The majority of the acid portion was comprised of citric acid (from 3.5 to 5.75 g 100 g⁻¹ DM) and lactic acid (from 2.5 to 7.3 g 100 g⁻¹ DM). The total acid content varied from 8.2 to 12.2 g 100 g⁻¹ DM. Lactic acid is produced by active cultures prior to pasteurization of whey; the longer the whey remains at elevated temperatures with active cultures, the greater the lactic acid content. With a glass transition temperature less than -50 °C, lactic acid is likely to have an enormous impact on drying properties of whey and DLP. Citric acid is used during whey processing to adjust the pH to less than 5.6 to enhance reverse osmosis. Its effects on drying of DLP are unknown.

The mineral content in the DLP samples varied from a low of 9.3 g 100 g⁻¹ DM to a high of 19.9 g 100 g⁻¹ DM, with substantial concentrations of magnesium, phosphorous, potassium, sodium and chloride ions. All DLP samples showed very low protein content, in the range of 1.36–2.37 g 100 g⁻¹ DM. The total solids in Table 1 do not add up to 100%, indicating that some minerals and non-protein nitrogen elements were not accounted for in the composition analysis.

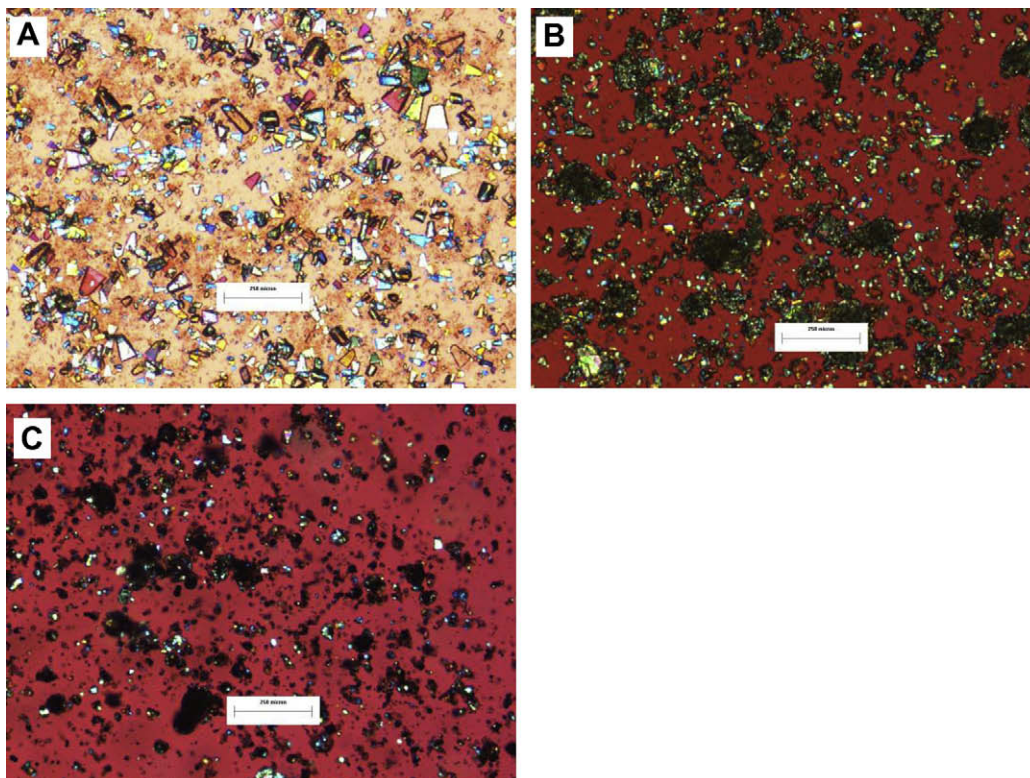


Fig. 1. Polarized light microscope images of delactosed permeate (DLP) samples: A, DLP 1 as obtained from industry; B, DLP 1 dried over silica gel; C, DLP 1 spray dried.

3.2. Characterization of state of various forms of DLP samples

Microscopy revealed that each of the DLP samples procured from industry had lactose in crystallized form, as shown for DLP 1 in Fig. 1A. After being dried over silica gel according to the protocol used for sorption kinetics in this study, crystalline material is clearly seen in DLP 1 (Fig. 1B), although with different size distribution than in the original material. The crystalline matter in the sample is comprised mostly of lactose, although the presence of crystalline salts cannot be ruled out. For comparison purposes, Fig. 1C shows the microscopic image of a spray dried DLP 1 (Unpublished). Both dried samples contain crystalline matter, although further analyses would be necessary to completely distinguish their physical state.

The XRPD patterns of DLP samples dried over silica gel (Fig. 2) suggest that all three samples consisted primarily of crystalline material, since no amorphous hump was discovered. All samples contained crystalline lactose, along with some other crystalline material (most likely salts).

3.3. Moisture sorption

The moisture sorption isotherms at 23 °C are shown in Fig. 3. For all DLP samples, two distinct regions of moisture uptake were observed. In the first region, large changes in the water activity, from 0.1 to 0.6, occurred with relatively small changes in moisture content. In the second region, above water activity of 0.6–0.7,

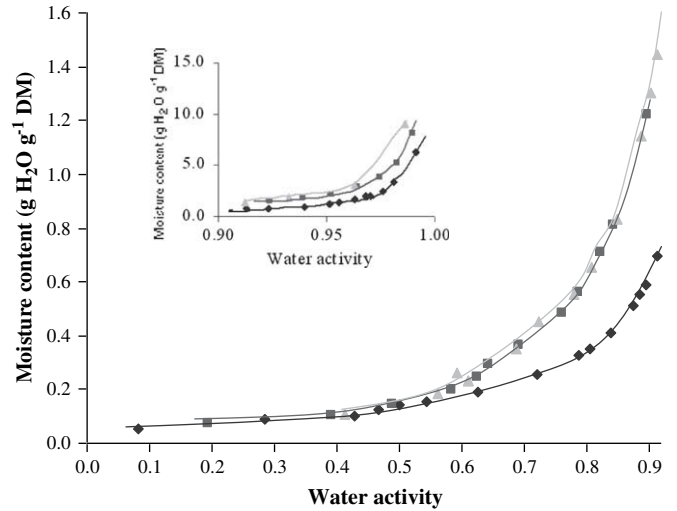


Fig. 3. Moisture sorption isotherms of delactosed permeate (DLP) samples at 23 °C: ◆, DLP 1; ■, DLP 2; ▲, DLP 3.

moisture content showed a sharp increase. At a water activity of 0.9, DLP samples 1, 2 and 3 had significantly different moisture contents of 0.62, 1.21 and 1.40 g H₂O g⁻¹ DM, respectively. The distinct regions observed for sorption isotherms could be attributed to the different modes of moisture uptake. At lower water activity, the water is strongly associated with the solutes in the DLP; whereas, above water activity of 0.6 there is a sharp increase in bulk-phase water (Fennema, 1985; Hardy, Scher, & Banon, 2002). At water activity above 0.6, DLP 1 showed the least moisture content compared with DLP 2 and 3. DLPs 2 and 3 had very similar equilibrium moisture content at respective water activity.

The kinetics of moisture sorption are shown in Fig. 4. Moisture uptake was the fastest in DLP 3 followed by DLP 2 and 1. DLP 3 also appeared to have the highest water content compared with others, as configured by the inset of Fig. 3 at high a_w. At any moisture content level above 0.19 g H₂O g⁻¹ DM, DLP 3 had a lower equilibrium water activity compared with other DLP samples leading to a higher driving force (environment a_w (1.0) – DLP equilibrium a_w) (Fig. 3), resulting in a higher moisture uptake rate compared with the others.

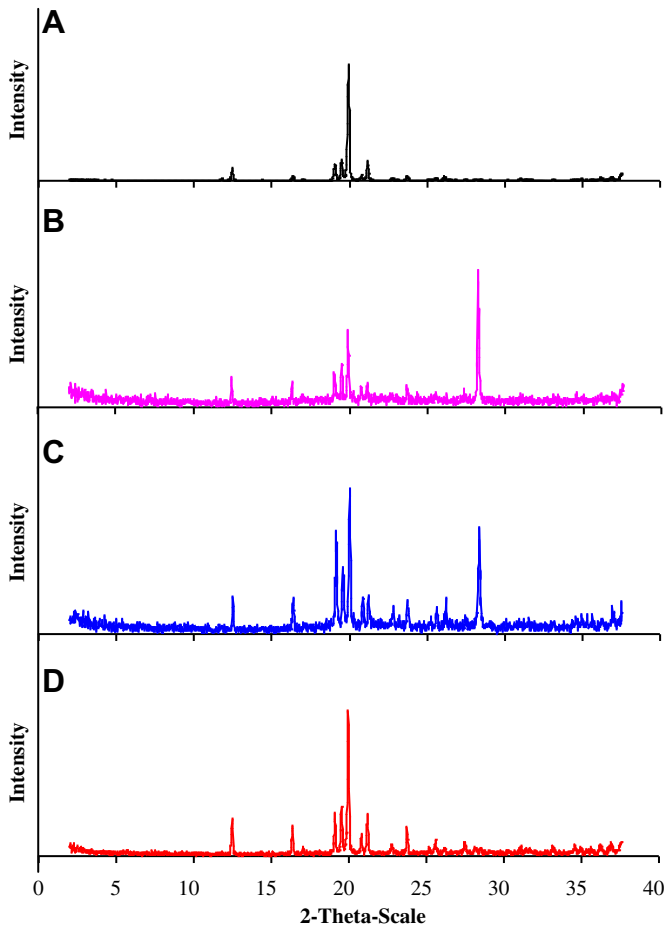


Fig. 2. X-ray powder diffraction (XRPD) patterns of delactosed permeate (DLP) samples dried over silica gel: A, lactose monohydrate; B, DLP 3; C, DLP 2; D, DLP 1.

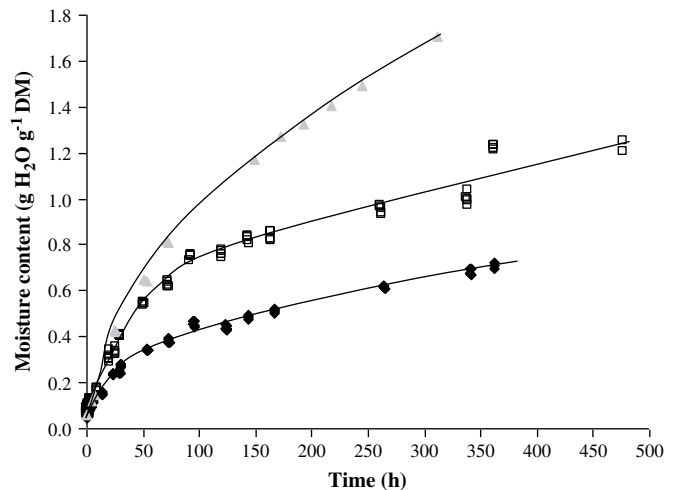


Fig. 4. Kinetics of moisture sorption of delactosed permeate (DLP) samples at 23 °C: ◆, DLP 1; □, DLP 2; ▲, DLP 3.

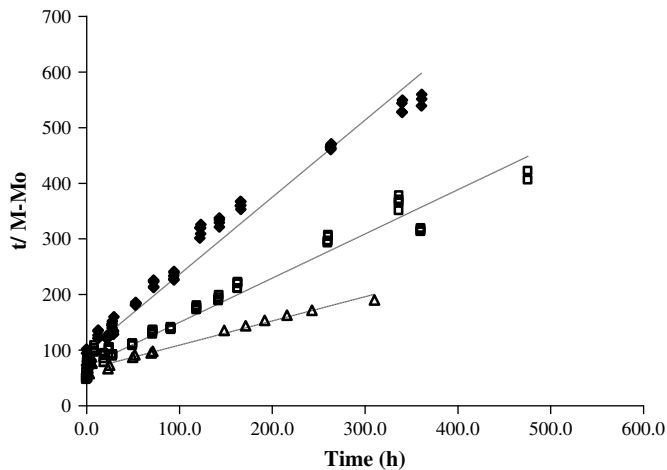


Fig. 5. Fitting of Peleg model (Eq. (1)) to moisture sorption data at 23 °C for delactosed permeate (DLP) samples: ◆, DLP 1; □, DLP 2; △, DLP 3.

3.4. Application of the Peleg model

The Peleg model was used to fit the DLP moisture sorption data at 23 °C to quantify the moisture uptake rate and moisture sorption capacity for individual DLP. The model fit resulted in $R^2 > 0.97$ for all DLP samples as shown in Fig. 5. The statistical analysis using SAS software was also carried out to investigate the goodness of fit of the Peleg model and also for obtaining approximate 95% confidence limits. The results are shown in Table 2. The P -value for all three DLP samples was observed to be <0.0001 , which indicated a good fit of the model to the sorption data.

The values for the Peleg constants K_1 and K_2 for DLP samples 1, 2 and 3 are shown in Table 2. K_1 is a constant related to mass transfer rate. Lower K_1 values indicate faster initial water sorption rate. K_1 value was highest in the case of DLP and was lowest in the case of DLP 3. This implied that the initial sorption rate was in the order of highest in DLP 3, followed by DLP 2 and 1.

K_2 is a constant related to maximum water absorption capacity (lower K_2 value indicates higher water sorption capacity). Again the trend observed for the K_2 values was similar to that of K_1 values. This implied that the moisture sorption capacity was considerably different for each of the DLP samples and decreased in the order from DLP 3, followed by DLP 2 and 1 (as confirmed by Fig. 4). The specific trend observed for the K_1 and K_2 values was thought to be because of the influence of certain compositional elements in DLP samples. Thus, the relationships between the Peleg constants (which represent the sorption behavior) with the compositional elements of DLP were investigated.

Table 2

Evaluation of the Peleg model (Eq. (1)) fit with the sorption data of delactosed permeate (DLP) samples.

Sample	Peleg constants	Approximate 95% confidence limit			P -value
DLP 1	K_1^a	94.39	86.86	101.9	<0.0001
	K_2^b	1.39	1.33	1.44	
DLP 2	K_1^a	68.00	62.87	73.14	<0.0001
	K_2^b	0.79	0.76	0.83	
DLP 3	K_1^a	63.19	57.78	68.60	<0.0001
	K_2^b	0.44	0.40	0.47	

^a K_1 value is expressed as $h \times (\text{g H}_2\text{O g}^{-1} \text{DM})^{-1}$.

^b K_2 value is expressed as $(\text{g H}_2\text{O g}^{-1} \text{DM})^{-1}$.

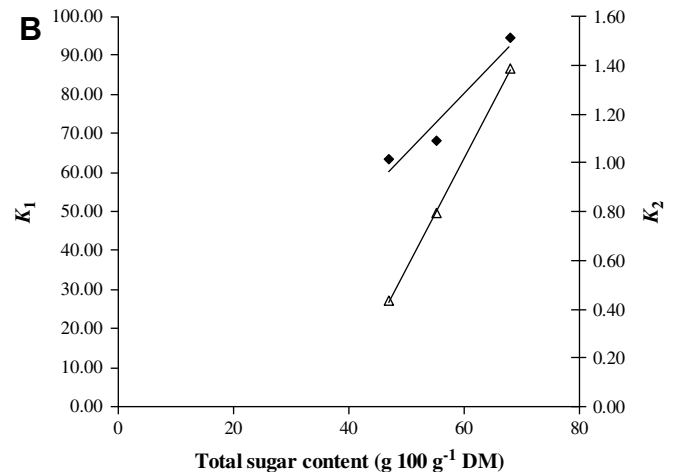
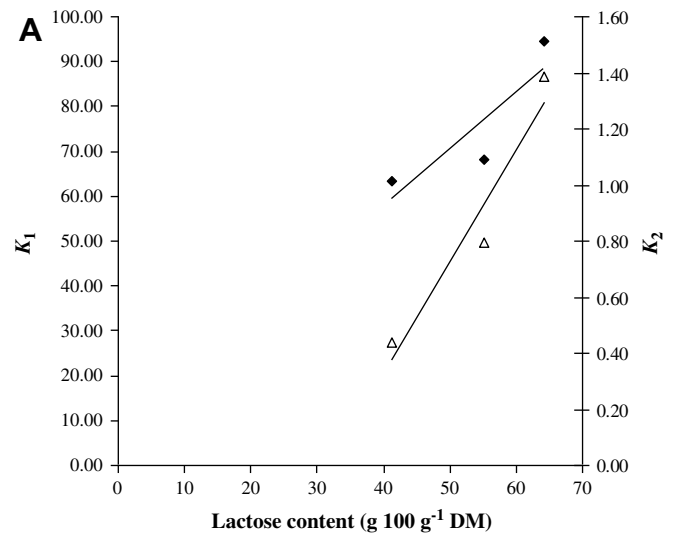


Fig. 6. Relationship between Peleg constants K_1 (◆) and K_2 (△) with: A, lactose; B, total sugars.

3.5. Relationship between the compositional elements of DLP and Peleg's constants

In the case of lactose and total sugars, K_1 and K_2 increased with an increase in concentration of lactose and total sugar (Fig. 6A and

Table 3

Relationship of Peleg's constants (K_1 and K_2) with constituents of delactosed permeate (DLP) samples.

DLP constituents	Constants	R^2	P -value ^{a,b}
Lactose	K_1	0.7672	0.32
	K_2	0.9266	0.17
Total sugars	K_1	0.9356	0.16
	K_2	0.9992	0.017
Citric acid	K_1	0.4575	0.53
	K_2	0.2415	0.67
Lactic acid	K_1	0.9672	0.11
	K_2	0.9978	0.03
Total acids	K_1	0.6141	0.42
	K_2	0.8177	0.28
Total minerals	K_1	0.9854	0.077
	K_2	0.9883	0.069

^a The significance level for P -value was set at $\alpha \approx 0.1$.

^b The values in bold text are statistically significant.

B). However, the regression analysis suggested that the relationships of lactose with K_1 and K_2 were not as significant (P -value > 0.1) as those of total sugars (P -value \leq 0.1) (Table 3). In particular the relationship of K_2 with total sugar content was highly significant (P -value = 0.017), which indicated that sugars other than lactose most likely also influence the sorption capacity in DLP samples, although no specific correlations with glucose and galactose were found.

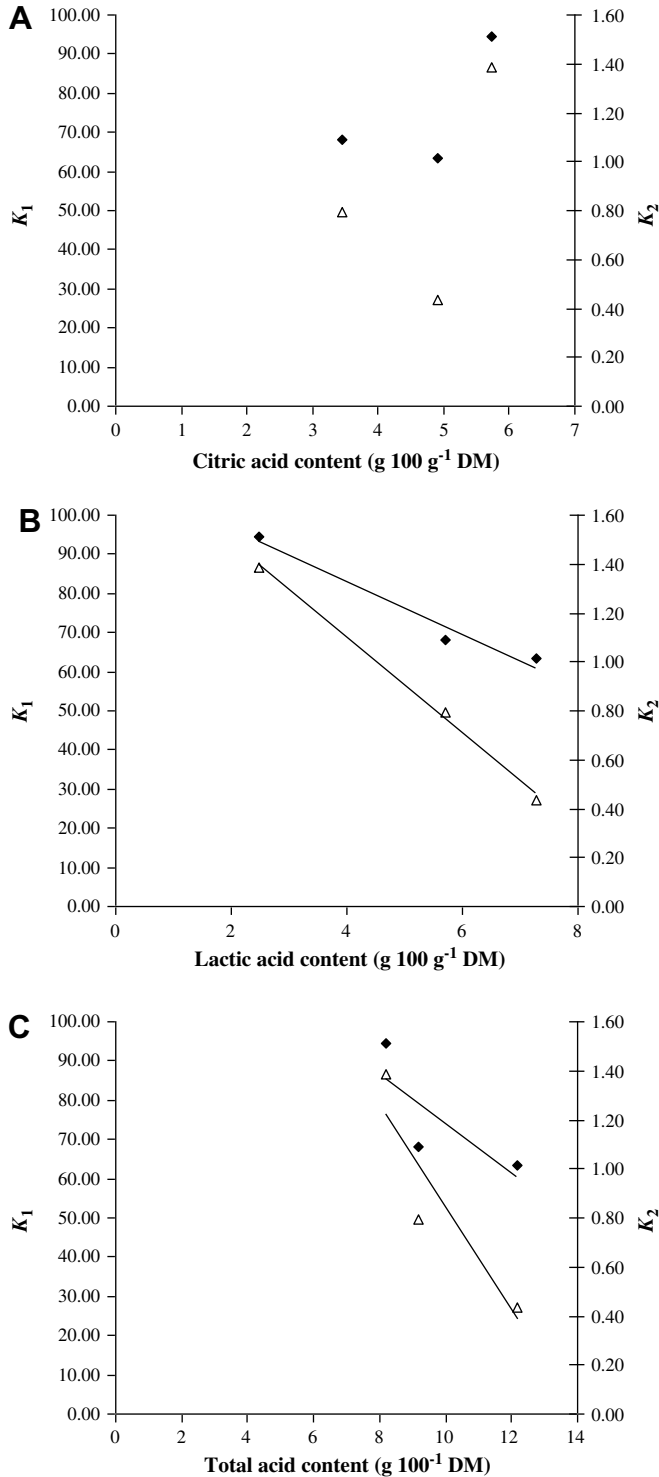


Fig. 7. Relationship between Peleg constants K_1 (◆) and K_2 (△) with: A, citric acid; B, lactic acid; C, total acids.

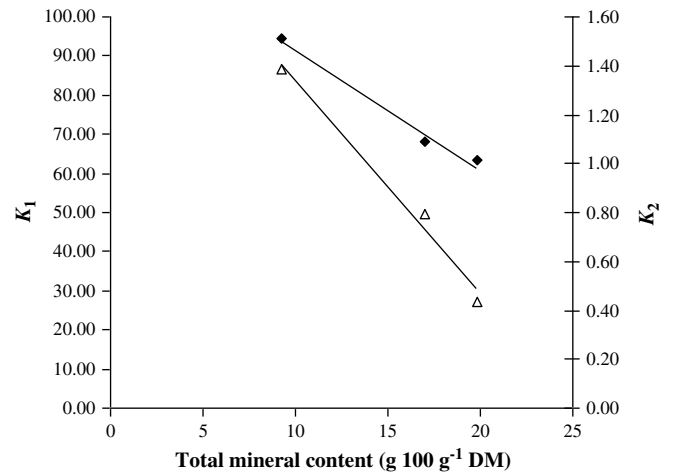


Fig. 8. Relationship between Peleg constants K_1 (◆) and K_2 (△) with minerals.

Citric and lactic acids were the majority contributors towards the total organic acid level in DLP. In the case of citric acid, no specific trend was observed with K_1 and K_2 (Fig. 7A). However, in the case of lactic acid, the K_1 and K_2 decreased with an increase in lactic acid content (Fig. 7B), with a high degree of significance (P -value \leq 0.1) (Table 3). This trend indicated that the initial rate of sorption and sorption capacity of DLP samples increased with an increase in the lactic acid content. For total acid content, the relationships with K_1 and K_2 were not very significant (Fig. 7C) (P -value > 0.1) (Table 3). This study suggested that among the total acids, only lactic acid had substantial influence on the sorption capacity of DLP samples.

In the case of minerals, the K_1 and K_2 decreased with an increase in total mineral content (Fig. 8). Similarly, the regression analysis also suggested that the relationship of total minerals with K_1 and K_2 was highly significant (P -value \leq 0.1) (Table 3). Based on the regression analysis of individual components of minerals, potassium (P -value – K_1 (0.1); K_2 (0.03)), sodium (P -value – K_1 (0.10); K_2 (0.25)) and chloride (P -value – K_1 (0.06); K_2 (0.21)) appeared to be the most important components for the observed relationship of minerals with K_1 . This suggested that with an increase in the mineral content, the initial sorption rate and sorption capacity of DLPs would increase. In an earlier study on protein concentrates, similar results were observed where the bound water content increased with an increase in salt concentration (Berlin, Kliman, Anderson, & Pallansch, 1973). In the present study, DLP 3 showed the highest initial sorption rate (K_1) and sorption capacity (K_2) among all the DLP samples. DLP 3 also had the highest lactic acid and mineral content (highest potassium, chloride content and sodium content close to highest) and lowest sugar content. In contrast, DLP 1 showed the lowest initial sorption rate (K_1) and sorption capacity (K_2); it also had the lowest lactic acid and mineral content, and the highest sugar content.

4. Conclusions

Delactosed permeate (DLP) is an effluent generated after separation of proteins and lactose from cheese whey. The present study reports the compositional analysis of DLP samples from different dairy processing industries and their sorption behavior. Significant differences in organic acid content were observed, most likely due to differences in whey pasteurization practices (lactic acid produced from live cultures) and membrane treatment systems (citric acid). Differences in lactose and mineral content observed

among the DLP samples could be related to different efficiencies of lactose removal in the refining operation. These compositional differences lead to differences in drying and sorption behavior.

Microscopy and XRPD analysis suggested that the DLP samples dried over silica gel, which were used to study the moisture sorption kinetics, consisted primarily of crystalline matter. The XRPD patterns documented the presence of crystalline lactose, along with other crystalline material (probably salts). More study would be required to confirm the state of individual constituents in the samples.

The Peleg model was observed to fit well to the sorption kinetic data at 23 °C for the DLP samples studied. The Peleg constants for initial sorption rate (K_1) and sorption capacity (K_2) obtained were correlated with the individual components of DLP samples in an attempt to identify the most influencing constituents towards the observed sorption behavior. The relationships between total sugar content (along with lactose content), lactic acid and minerals (especially potassium, sodium and chloride) were found to be the most significant and influential to the sorption behavior of DLPs. With an increase in the lactic acid and mineral content the initial sorption rate and the sorption capacity of DLPs were found to increase significantly.

Although DLP samples in this study were dried over silica gel rather than by spray drying (as would be commercially practiced), differences in sorption behavior observed here are likely to correlate to differences in properties of commercially processed materials as well. Further work to better understand the properties of

DLP that most significantly impact drying and moisture sorption are warranted.

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