HPLC and NMR Study of the Reduction of Sweet Whey Permeate

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Sweet whey permeate was hydrogenated under high pressure and temperature with Raney Ni as a catalyst. Different reaction conditions, such as reaction time, amount of catalyst, and initial hydrogen pressure and temperature, were studied. The reaction mixtures were analyzed by HPLC and NMR. The major whey component, lactose (4-*O*-*β*-D-galactopyranosyl-D-glucose), was converted under these conditions to lactitol (4-*O*-*β*-D-galactopyranosyl-D-glucitol) to different degrees. Hydrolysis of lactose to galactose and glucose and further reduction was also observed over a wide temperature range, whereas isomerization of lactose to lactulose (4-*O*-*β*-D-galactopyranosyl-D-fructose) and subsequent reduction only occurred above 110 °C. With reaction time of 4 h, an initial hydrogen pressure of 1500 psi, 40.5 g of Raney Ni, and a temperature = 120 °C, sweet whey permeate (42%, 340 g) gives lactitol (85.2 %), lactulitol (1.7%), and sorbitol and dulcitol (0.8%) together with unreacted fat, protein, and salts (12.3%).

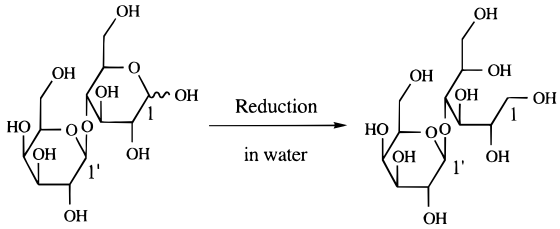
Keywords: *Sweet whey permeate; lactose; lactitol; isomerization; hydrolysis; lactulose; glucose; galactose; sorbitol; dulcitol; lactulitol; catalytic hydrogenation; HPLC; NMR*

INTRODUCTION

Worldwide production of whey is on the order of 130 million tons each year, and the United States accounts for nearly 21% of this production (Zall, 1992). As the byproduct of cheese manufacture, whey poses an environmental problem because of its high biochemical and chemical oxygen demand. Full utilization of whey (or whey permeate) and lactose, the major component in whey permeate, has not yet been realized. Current applications include whey and lactose used for ice cream, frozen desserts, bakery products, beverages, and meat products; whey as an additive in animal feed; and lactose as an inert carrier for medical pills (Moor, 1992). Schwartz (1987) also demonstrated that xanthan gum made from whey or lactose exhibited excellent viscosity properties compared with those of commercial xanthan gum. Recently, industry has paid more attention to the utilization of other lactose derivatives, for example, lactulose (Mizota et al., 1987), lactosyl urea (McAllan et al., 1975), and lactitol. Among these derivatives, lactitol is of particular interest because it has wider applications than lactose. In 1993, the FDA ruled that lactitol can be used in the U.S. as a sugar substitute. Lactitol provides less sweetness and fewer calories, and also is suitable for diabetics. Many food applications, such as chocolates, candies, caramels, fondants, chewing gum, frozen deserts, and bakery goods, use lactitol as a bulk sweetener (Blankers, 1995; Linko et al., 1980; Linko, 1982). Scholnick and Linfield (1977) demonstrated that surfactants can be made from alkoxylation of lactitol. In a recent study from our laboratory, lactitol was shown to be superior to lactose and sucrose for preparing polyurethane rigid foam because of its lack of reducing functionality and higher thermostability (Wilson and Hu et al., 1996).

Because of these diverse applications, the demand for lactitol production will increase in the coming years. Lactitol can be produced from lactose reduction (Scheme 1) either by $NaBH_4$ (Abdel-Akher et al., 1951) or catalytic hydrogenation (Konishi et al., 1990; Darsow, 1991; Ishii, 1988; Saijonmaa, 1978). Currently, industry uses the latter method to produce large quantities of food-grade lactitol via Raney Ni catalyst. Although catalytic reduction of pure lactose has been reported, lactitol production by the catalytic reduction of whey permeate (whey proteins are removed by ultrafiltration from whey) has not been explored in detail. Guidini et al. (1983) studied this topic, but pure lactitol could only be obtained from deionized whey permeate (at added cost of manufacture). In this paper, we report our study on the catalytic reduction of lactose in sweet whey permeate by Raney Ni catalysis under various reaction conditions.

Scheme 1. Reduction of Lactose to Lactitol by either $NaBH_4$ or Catalytic Hydrogenation



lactose (**1**) lactitol (**2**)

MATERIALS AND METHODS

Materials. α-Lactose monohydrate, D-glucose, D-galactose, sorbitol, dulcitol and Raney Ni were purchased from Aldrich Chemical Company (Milwaukee, WI); lactulose was purchased from Sigma Chemical Company (St. Louis, MO); lactitol monohydrate was a free gift from Purac America, Inc. (Lincolnshire, IL); dry whey permeate (product code 396, with a composition listed in Table 1), was kindly provided by Foremost Ingredient Group (Baraboo, WI). Deionized water (filtered by Nanopure II system from Barnstead, Dubuque, IA) was used for both reduction and HPLC analysis.

General Procedures for Hydrogenation. All 42% (solid by weight) whey solutions were made by mixing 150 g of dry whey permeate with 190 g of deionized water. The amount of

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Table 1. Components of Dry Whey Permeate (Code 396, a Nonhydroscopic Spray-Dried, Lactose-Rich Product from Sweet Dairy Whey)

proximate analysis	typical wt %	proximate analysis	typical wt %
lactose	83.0	ash	8.5
protein	3.5	water	4.8
fat	0.2		

Raney Ni (used as received) was measured as follows. The Ni/water slurry was transferred into a test tube, and the Ni was allowed to settle to the bottom. The top water layer was transferred back to the bottle and more Ni/water slurry was added to the test tube until the desired amount of Raney Ni was measured.

Whey permeate solution and catalyst were transferred into a 1-L glass sleeve with good mixing, and the pH was measured by a microprocessor pH/millivolt meter 811 (Orion Research Inc., Boston, MA) at ambient temperature. The sleeve was then placed into a 1-L autoclave (Autoclave Engineers, Erie, PA), sealed, pressurized at 500 psi, and purged with nitrogen three times; the final pressurization lasted 30 min to detect leaks. Hydrogen gas was introduced into the autoclave to a desired pressure (initial hydrogen pressure, P_i). The reaction was ramped to the highest temperature (T_{set}) at a rate of 1.6 °C/min and allowed to react with stirring at 750 rpm. After the designated time (including ramping time), the reaction was stopped by cooling the system to 50 °C, which was followed by releasing the pressure and unsealing. An aliquot of the crude product was centrifuged in a table centrifuge (IEC Clinical Centrifuge, International Equipment Company, Needham Hts., MA) at a rate of 15 000 rpm. The aqueous solution was then collected for analysis.

Reduction of Lactulose. We observed isomerization of lactose to lactulose, so reduction of pure lactulose was independently studied. Lactulose was hydrogenated as described for whey permeate under high pressure and temperature with Raney Ni for 14 to 24 h to ensure complete reduction.

NMR Analysis. All NMR analyses were run with a GE-300 instrument (General Electric, NMR-Instruments, Fremont, CA). For ^{1}H NMR, the sodium salt of 3-(trimethylsilyl)-1-propanesulfonic acid (DSS) was used as internal standard. For ^{13}C NMR, dioxane was used as internal standard. D_2O was used as solvent.

HPLC Analysis. For HPLC analysis (IBM LC/9533, IBM Instruments Inc., San Jose, CA), a 20-μL sample (0.2 to 0.3 mg/mL) was injected at a 0.5 mL/min flow rate, with pure water as eluent. The samples were first passed through a Coregel-87C calcium form guard column and then through a Coregel-87C calcium form analytical column (Interaction Chromatography, San Jose, CA) for separation at both 25 and 85 °C. The higher temperature was controlled by a Waters Temperature Control Module (Millipore Waters Chromatography, Marlborough, MA). The separated samples were detected by a differential refractometer (Knauer, Rainin Instrument Company, Emeryville, CA) and analyzed with a HP 3990 integrator. The deionized water was deaerated and filtered through 0.2-μÅ HPLC filter paper.

RESULTS AND DISCUSSION

Retention Time. The separation of lactose, lactulose, lactitol, galactose, and glucose is difficult. One reported method used Lichrosorb-NH_2 and Lichrosorb-Si-60 columns (Guidini et al., 1983) to separate these sugars, but separation was poor. Cationic ion-exchange resin has also been used to separate lactose, lactulose, and galactose (Dendene et al., 1995). In this paper, we used a Coregel-87C column that contains a calcium form of sulfonated polystyrene ion-exchange resin cross-linked with divinylbenzene to separate these sugars. This column combines the properties of adsorption, complexation, and size-exclusion.

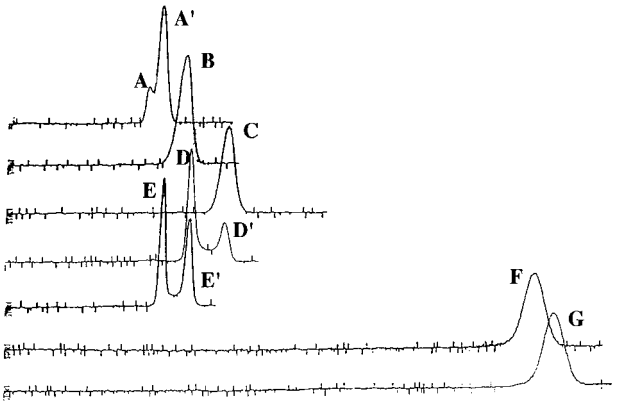


Figure 1. HPLC chromatogram of sugars separated by Coregel-87C. Retention times (R_t) determined at ambient temperature (0.3 mg/mL concentration, 0.5 mL/min flow rate). (A) β-lactose (R_t = 12.40); (A′) α-lactose (R_t = 13.52); (B) lactulose (R_t = 15.46 min); (C) lactitol (19.57 min); (D) β-galactose (R_t= 16.44 min); (D′) α-galactose (19.48 min); (E) β-glucose (13.90 min); (E′) α-glucose (16.24 min); (F) dulcitol (49.15 min), and (G) sorbitol (50.09 min).

Table 2. Sugar Retention Times at Different Concentrations When Separated by Coregel-87C (0.5 mL/min Flow Rate) at 85 °C

	retention time (min)	
sugar	0.3 mg/mL	0.6 mg/mL
lactose	9.87	10.23
lactulose	10.76	11.24
lactitol	12.95	13.52
galactose	13.00	13.45
glucose	11.54	12.09
dulcitol	23.28	24.10
sorbitol	24.34	25.35

Typical retention times of lactose, lactulose, lactitol, galactose, glucose, dulcitol (galactitol), and sorbitol (glucitol) at ambient temperature are shown in Figure 1. At this temperature, α- and β- anomers of reducing sugars such as lactose, glucose, and galactose, can be separated, whereas lactulose, lactitol, dulcitol, and sorbitol show only one peak. α-Lactose overlapped with β-glucose, lactulose overlapped with β-galactose and α-glucose, and lactitol overlapped with α-galactose. The retention times for dulcitol and sorbitol were ∼1 h. These characteristics presented difficulties for analyzing the products from reduction of whey permeate. The overlapping and long retention times could be overcome by running the HPLC at high column temperature. At 85 °C, the interconversion rate of α- and β- anomers increased, resulting in one HPLC peak. All sugars had shorter retention times, especially dulcitol and sorbitol, and lactitol could be completely separated from lactose, lactulose, glucose, sorbitol, and dulcitol. Although lactitol and dulcitol showed very close retention times, they could be differentiated by ^{13}C NMR because dulcitol gives two distinct peaks at 63.18 and 70.10 ppm.

Not only temperature but also concentration can affect the retention times of these sugars (Table 2). Lower concentration tends to affect nonreducing sugar more than reducing sugar (e.g., ΔR_t for sorbitol is 1 min, whereas that for lactose it is only 0.36 min at 0.3 and 0.5 mg/mL). We therefore strictly controlled the sample concentration and column temperature in all HPLC analyses.

Calibration of the Column and Refractive Index of the Sugars. The HPLC column was calibrated with pure lactose solution at an 85 °C column temperature. Good linearity was obtained in concentrations ranging

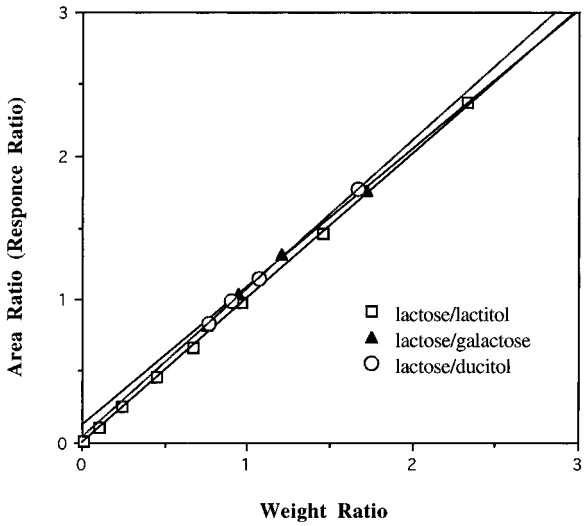


Figure 2. Selected internal standard curves based on lactose/lactitol, lactose/galactose, and lactose/dulcitol. The concentration of lactose solution is from 0.024 to 2.44 mg/mL (85 °C column temperature, 0.5 mL/min flow rate).

from 0.024 to 2.488 mg/mL (corrected for water in lactose monohydrate). The peak area is directly proportional to the amount (weight) injected.

To measure the ratio of each component in reduced whey, internal standard curves of all sugars were constructed. The three curves shown in Figure 2 correspond to well-separated lactose/lactitol, lactose/galactose, and lactose/dulcitol. The linearity indicates that lactose, lactitol, lactulose, galactose, glucose, sorbitol, and dulcitol have similar refractive indexes (all curves are based on weight ratios instead of mole ratios and are corrected for hydration).

Reduction of Lactulose. Lactose can isomerize to lactulose both in alkaline media and by heat treatment (Adachi and Patton, 1961). Although sweet whey permeate is slightly acidic (42% of the solution has a pH of 5.8−6), lactose can still isomerize under high temperatures. Furthermore, lactulose can be reduced under the same conditions that would reduced lactose.

Lactulose was hydrogenated to lactitol and lactulitol [4-*O*-*β*-D-galactopyranosyl-D-mannitol] according to Guidini et al. (1983). To establish the retention time for lactulitol on Coregel-87C, 25 g of lactulose in 100 mL of Millipore water was hydrogenated at 120 °C for 14 and 24 h under pressure (P_i = 1500 psi) in the presence of 10 g of Raney Ni. The reducing product gave three peaks with a Coregel-87C column at 85 °C (Figure 3a); two major peaks at retention time (R_t) of 11.33 and 12.95 min and one minor peak at R_t of 12.17 min. The 12.95-min peak is clearly due to the formation of lactitol. The ^{1}H NMR of this mixture (Figure 3c) indicated no lactulose (Figure 3b) remained, but four products were formed (four pairs of doublets: 4.549/4.574, 4.487/4.512, 4.480/4.506, and 4.449/4.475 ppm). The 4.480/4.506 ppm doublet is due to the lactitol C-1′ proton (see Scheme 1). Other doublets may result from C-1′ protons of **7**, **8**, and **9**, which were reduced from lactulose (**3**), and its isomers lactose (**1**) and isolactulose (**6**), respectively under high temperature and pressure. The proposed mechanism is show in Scheme 3. The major products were lactulitol (R_t = 11.33 min) and lactitol (R_t = 12.95 min).

Reduction of Whey Permeate. To find optimum reaction conditions for reducing whey lactose, we first studied the conversion (by ^{1}H NMR analysis only) with

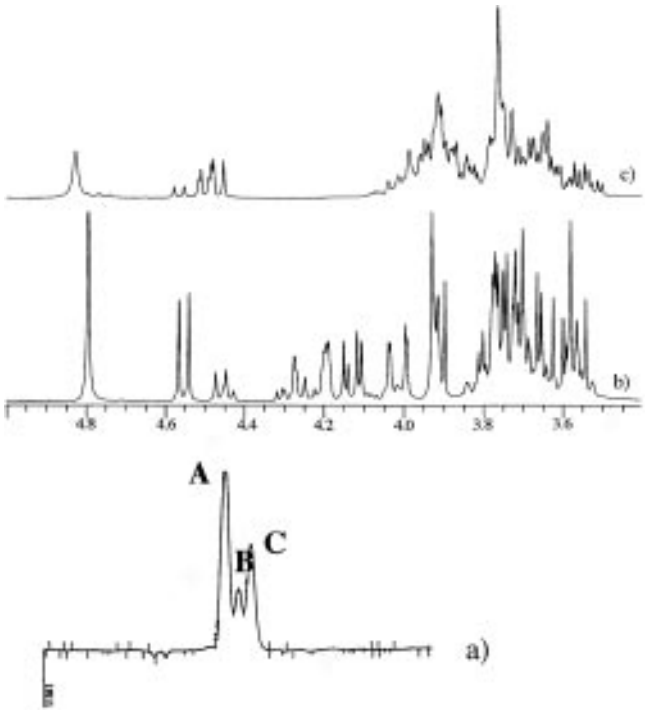
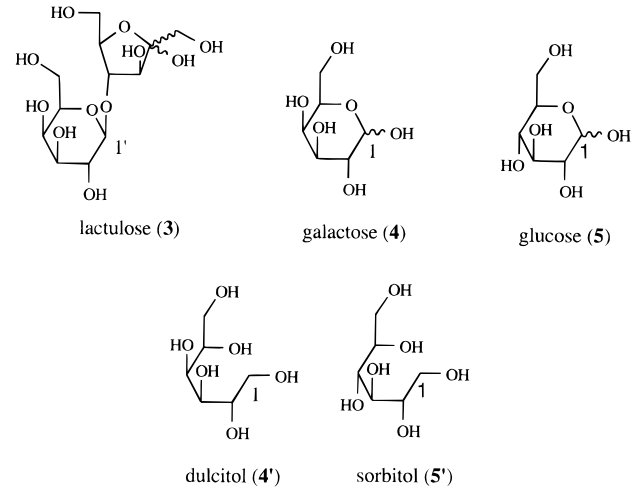


Figure 3. (a) HPLC chromatogram of reduced lactulose by Coregel-87C. Retention times (R_t) determined at 85 °C (0.5 mL/min flow rate). (A) Lactulitol (R_t = 11.33 min); (B) (R_t = 12.17 min), (C) lactitol (R_t = 12.95 min). (b) ^{1}H NMR of lactulose. (c) ^{1}H NMR of reduced lactulose.

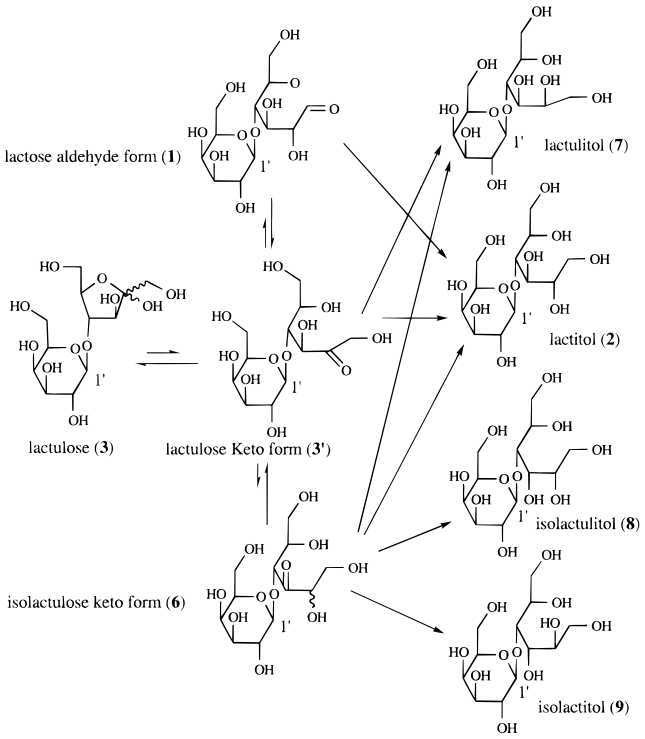
Scheme 2. Structures of Lactulose (3), Galactose (4), Dulcitol (4′), Glucose (5), and Sorbitol (5′)



different whey concentrations (12, 25, and 42%) and different weight ratios of solid whey permeate versus catalyst and temperature. Whey permeate lactose disappearance depended on the parameters chosen. At low whey solid/Raney Ni ratios, no lactose remained according to ^{1}H NMR [no peaks at 5.22, 5.21, 4.67, 4.63 ppm (α and *β* H at C-1 of lactose) and 4.46, 4.43 ppm (α H at C-1′ of lactose); see Scheme 1]. Higher temperature also improved conversion. With high whey solid/Raney Ni ratios (or less catalyst), the reaction did not go to completion.

Based on these observations and the conditions used to completely reduce pure lactose (Guidini et al., 1983), we selected 42% whey permeate solid as our model to study how reaction time, hydrogen pressure, amount of catalyst, and temperature affect whey lactose conversion. Another reason for choosing 42% whey permeate is that it is a close mimic of the concentrated whey

Scheme 3. Proposed Isomerizations and Reduction Products



permeate product ($0.42/pound) resulting from cheese manufacture.

Reaction Time versus Lactose Conversion. With a fixed concentration of whey permeate (42%), pH (42% whey with Raney Ni has a pH between 5.82 and 5.86 at ambient pressure and ambient temperature), initial hydrogen pressure (P_i = 1500 psi) at ambient temperature, highest equilibrium reaction temperature (T_{set} = 120 °C), and amount of Raney Ni (40.5 g), reaction time was increased from 1.5 to 10 h. The HPLC analysis of whey permeate before reduction is shown in Figure 4. In addition to 84.1% lactose, two impurities at 7.21 and 7.88 min were detected. Figure 4b is a typical HPLC chromatogram after the reduction showing unchanged impurities while hydrolysis, isomerization, and reduction of lactose occurred. The results from 1.5- to 10-h reaction times are depicted in Figure 5.

For a reaction time of 1.5 h, isomerization of lactose to lactulose was not observed. Only reduction and hydrolysis of lactose had occurred. The hydrolysis products, galactose and glucose, were also reduced to dulcitol and sorbitol, respectively, since no anomeric protons (see anomeric protons of both galactose and glucose in Scheme 2) from either galactose or glucose were present in the ^{1}H NMR spectra. The formation of dulcitol can also be verified by two characteristic ^{13}C NMR peaks at 63.18 and 70.10 ppm. At 1.5 h, lactitol (55.9%) and lactose (29.2%) are present. As reaction time increased to 2 h, isomerization occurs (HPLC peaks at R_t of 10.76 min represents 3% lactulose formed during the reaction), but without any reduced lactulose products formed. This result indicates that lactulose is much more difficult to reduce than lactose. On the other hand, the amounts of dulcitol and sorbitol remained unchanged. At reaction times of 2.5 and 3 h, the amounts of lactulose and the reduced hydrolyzed monomers remained unchanged, but lactose decreased and lactitol increased. At 4 h, lactose had completely disappeared and lactulose was partially reduced. After

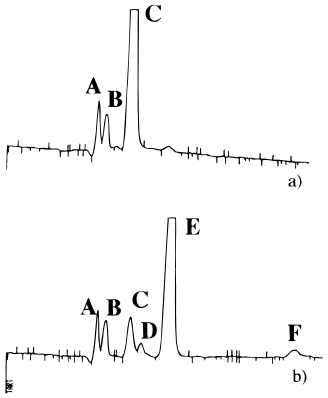


Figure 4. HPLC chromatogram of (a) whey permeate and (b) a typical reduced whey permeate by Coregel-87C. Retention times (R_t) were determined at 85 °C (0.5 mL/min flow rate). (A) Impurity 1 (R_t = 7.21 min); (B) impurity 2 (R_t = 7.88); (C) lactose (R_t = 9.84 min); (D) lactulose (R_t = 10.72 min); (E) lactitol (R_t = 12.93 min); (F) dulcitol and sorbitol.

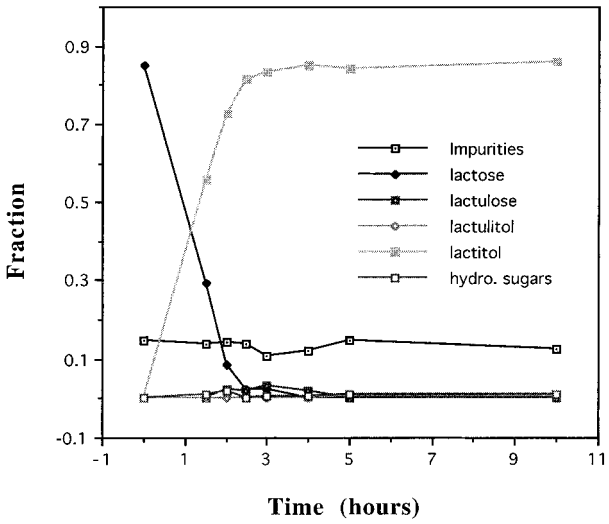


Figure 5. Fraction of each component resulting from reduction of 42% whey permeate (340 g, pH 5.82−5.86) by 40.5 g of Raney Ni at P_i = 1500 psi (at ambient temperature), with the highest equilibrium temperature (T_{set}) of 120 °C.

5 h, lactulose was completely reduced and HPLC showed a lactulitol peak at 11.34 min. Extension of the reaction time to 10 h gave similar results to that of 5 h, with no extra hydrolyzed products being formed. This result implies that lactitol is more stable to hydrolysis than lactose (Donnelly, 1991). The trend for each component from 1.5 to 10 h is shown in Figure 5. On the whole, hydrolyzed sugars account for only 2% of the total weight.

Catalyst Amount versus Lactose Conversion. Based on the reaction time study, we fixed the reaction time at 3 h, initial hydrogen pressure at 1500 psi (at ambient temperature), and highest equilibrium reaction temperature (T_{set}) at 120 °C, and varied the amount of Raney Ni to study its effect on the reduction of 42% whey permeate. The trends for each component with five different amounts of catalyst are shown in Figure 6. When a lower amount of catalyst was used, a higher amount of isomerization of lactose to lactulose occurred. For 15 g of Raney Ni, ~6% of lactulose was detected. For 20 g of Raney Ni, ~4.4% lactulose was detected, and so on. The amounts of dulcitol and sorbitol were also higher for lower amounts of catalyst (up to 4% when 15 g of Ni was used). When 30 g of Ni was used, most of the lactose was converted to lactitol, and only 4% by

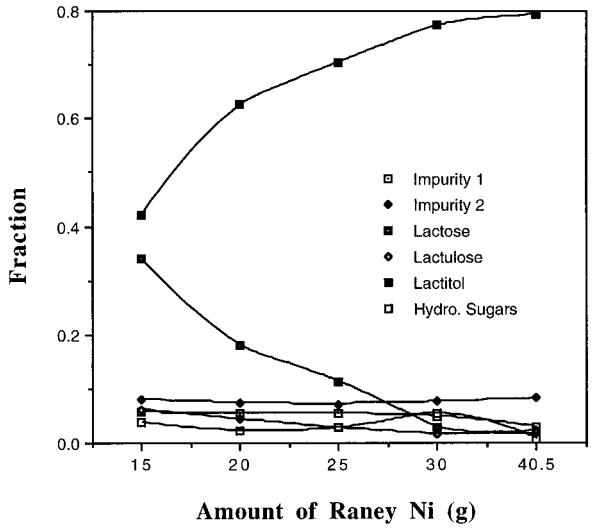


Figure 6. Fraction of each component resulting from reduction of 42% whey permeate (340 g, pH 5.82−5.86) by various amounts of Raney Ni with P_i = 1500 psi (at ambient temperature), T_{set} = 120 °C, and 3 h of reaction time.

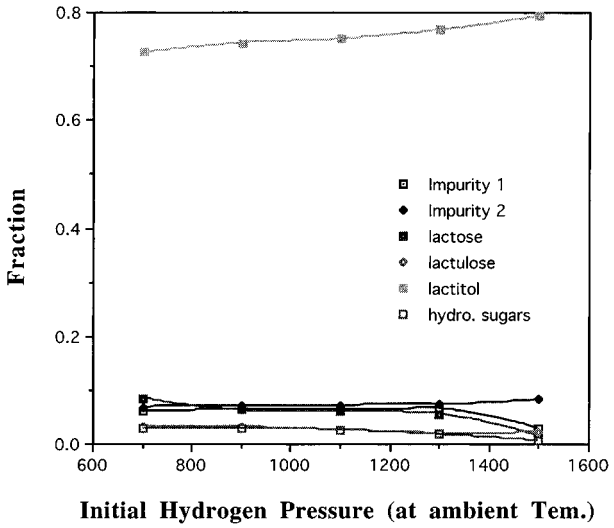


Figure 7. Fraction of each component from reduction of 42% whey permeate (340 g, pH 5.82−5.86) by 40.5 g of Raney Ni, with various P_i (at ambient temperature), highest equilibrium temperature (T_{set}) of 120 °C, and 3 h of reaction time.

weight of lactose remained. An increase of reaction time for this catalyst amount led to complete conversion of lactose. Thus, less catalyst and longer reaction time will afford complete conversion of lactose.

Hydrogen Pressure versus Lactose Conversion. The third parameter studied was the initial hydrogen pressure. Having confirmed that 40.5 g of catalyst and 3 h reaction time under 1500 psi H_2 pressure with T_{set} of 120 °C will leave only 2% of unreacted lactose, lower initial pressure was used to evaluate its effect on lactose conversion.

Pressure does not affect reaction components to the extent that reaction time and amount of catalyst do, as shown in Figure 7. At P_i = 700 psi, lactose accounts for 8.5% of total solid weight, whereas lactitol accounts for 72.5%. At P_i = 1500 psi, only 2% of lactose remains, but lactitol reached 79.3%. Isomerization and hydrolysis remain unchanged through this pressure range.

From the data in Figure 5, 6, and 7, we can conclude that at defined temperature and pH (T_{set} = 120 °C and pH 5.8), the amount of catalyst can affect the

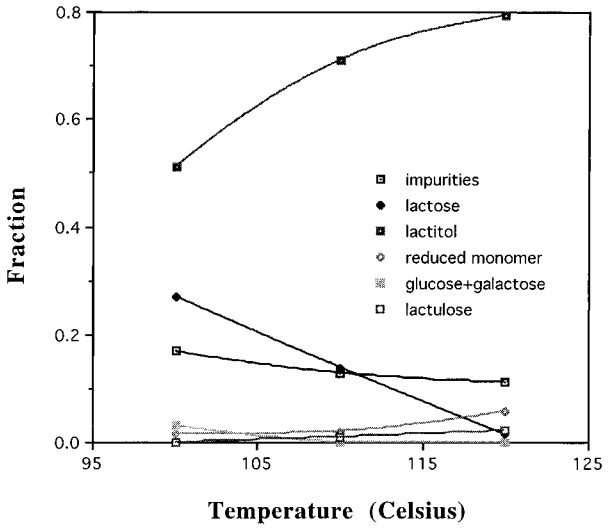


Figure 8. Fraction of each component from reduction of 42% whey permeate (340 g, pH 5.82−5.86) by 40.5 g of Raney Ni, with P_i = 1500 psi (at ambient temperature), various of highest equilibrium temperatures (T_{set}), and 3 h of reaction time.

isomerization and hydrolysis of lactose. Pressure and reaction time are less important for these two side reactions.

Temperature versus Lactose Conversion. The last important parameter that can affect the reduction of whey permeate is temperature. Three different temperatures (100, 110, and 120 °C) were chosen for this study. Reduction of lactose takes place much faster at higher temperature (120 °C; Figure 8). At 100 °C, only ~51% of lactitol was formed from lactose, and hydrolyzed glucose and galactose were partially reduced to sorbitol and dulcitol. An HPLC peak at 11.54 min indicated the existence of glucose (confirmed by ^{1}H NMR analysis). Lack of a lactulose peak at 10.76 min also indicated that lactulose is not formed at 100 °C in 3 h.

When T_{set} was 110 °C, lactulose was detected by HPLC analysis, but only in a small quantity (~1%). The hydrolyzed glucose and galactose were completely reduced, and the amount of lactitol increased by 40%. Increasing T_{set} to 120 °C only increased the lactulose produced to 2% of total weight. Hydrolysis also increased at higher temperature, but to a lesser extent. Hence, we can conclude that temperature affects not only the rate of reduction of lactose but also the rate of isomerization and hydrolysis of lactose.

CONCLUSIONS

We found that whey permeate lactose can be reduced to lactitol under proper conditions, but minor side reactions do occur. The isomerized and/or hydrolyzed products depend on the reaction parameters of time, hydrogen pressure, catalyst amount, and reaction temperature. Optimal conditions for lactose to lactitol reduction are reaction time = 4 h, initial hydrogen pressure = 1500 psi, a dry whey permeate/Raney Ni ratio of 3.7:1 (w/w), and temperature = 120 °C. Under these conditions, sweet whey permeate gives lactitol (85.2%), lactulitol (1.7%), sorbitol + dulcitol (0.8% combined), together with unreacted fat, protein, and salts (12.3%). Lactitol hydrate can be crystallized from the reduced whey permeate syrup (12.7% water) upon cooling to 0 °C.

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