Porosity of Microcapsules with Wall Systems Consisting of Whey Proteins and Lactose Measured by Gas Displacement Pycnometry

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ABSTRACT

Porosity characteristics of anhydrous milkfat-containing, spray-dried microcapsules with wall systems consisting of mixtures of whey proteins and lactose were investigated by gas displacement technique, using helium and nitrogen as displacing gases. Microcapsules exhibited molecular-sieve type porosity. Results indicated that 8–12% of microcapsule volume was inaccessible to helium. Permeation of nitrogen to wall matrices was inversely proportional to lactose content. Pore volume that was inaccessible to nitrogen penetration ranged from 0.128 to 0.263 cm^3/g for core-free and from 0.121 to 0.214 cm^3/g for core-containing capsules. It was affected by wall composition and core load. Results suggested that adjusting microcapsule composition could modulate gas penetration into microcapsules.

Key Words: microcapsules, porosity, whey proteins, lactose, gas permeation

INTRODUCTION

POROSITY CHARACTERISTICS ARE IMPORTANT TO MICROCAPSULE functionality. Wall matrix of microcapsules containing food ingredients is designed to control mass transfer between the core and the environment (Rosenberg, 1985; Dziezak, 1988; Jackson and Lee, 1991; Arshady, 1993; Shahidi and Han, 1993). This includes controlling permeation of gases (oxygen) and/or volatiles through the microcapsule wall. Permeability of wall matrix to oxygen is affected by porosity of the wall matrix and determines the oxidative stability of the core (Moreau and Rosenberg, 1996, 1998). With volatile core, wall permeability affects core loss during storage (Rosenberg, 1985).

Information about porosity of microencapsulated food ingredients is limited. Porosity characteristics of gum arabic-based, volatiles-containing, spray-dried microcapsules were reported by Rosenberg (1985). Microcapsules exhibited a molecular-sieve type porosity that was affected by composition, drying parameters and storage conditions. The term "molecular-sieve" type porosity describes porosity of materials with pores, small enough to exclude or resist permeation of molecules (Kärger and Ruthven, 1992). Rosenberg (1985) reported that volatile loss from gum arabic-based microcapsules (during storage) correlated well with porosity characteristics. The porosity of whey protein-based microcapsules containing anhydrous milkfat was studied by gas-displacement technique. They were reported to exhibit a molecular-sieve type porosity that was affected by wall solids concentration (prior to drying) and core load (Moreau and Rosenberg, 1998). About 10% of microcapsule volume was reported to be inaccessible to He permeation and pore volume accessible to N_2 permeation decreased with core load and wall solids concentration (Moreau and Rosenberg, 1998). The ratio between proportion of whey proteins adsorbed at the core surface to that forming the wall matrix affected porosity characteristics of microcapsules (Moreau and Rosenberg, 1998).

Wall systems consisting of whey proteins and lactose have been reported to be effective in microencapsulation of anhydrous milkfat (Young et al., 1993; Moreau and Rosenberg, 1993, 1996; Rosenberg, 1997). Core extractability in microcapsules consisting of mixtures of whey proteins and lactose was inversely related to proportion of lactose included in the wall and was affected by physical state of lactose (Moreau and Rosenberg, 1993; Young et al., 1993a; Rosenberg, 1997). Oxidative stability of anhydrous milkfat microencapsulated in whey protein-based wall systems was influenced by lactose content (Moreau and Rosenberg, 1996). Reports indicated effects of lactose on mass transfer through wall systems consisting of whey proteins and lactose. In light of the effects of porosity in governing mass transfer through wall matrices of microcapsules, the influence of lactose may, potentially, be related to effect of lactose content (in microcapsule) on wall porosity. Porosity of microcapsules with wall systems consisting of whey protein and lactose has not been reported. Understanding the effects of lactose on porosity of microcapsule wall matrices can provide means to design and control porosity characteristics for different applications.

The objective of our research was to investigate the porosity characteristics of spray-dried, anhydrous milkfat-containing microcapsules with wall systems consisting of mixtures of whey proteins and lactose.

MATERIALS & METHODS

Core and wall materials

Whey protein isolate (WPI) was purchased from Le Sueur Isolates (Le Sueur, MN). The WPI contained 95.4% protein, 1.84% ash, and 2.68% moisture. D-lactose was purchased from Sigma Chemical Company (St. Louis, MO). Anhydrous milkfat (AMF) with fat content of 99.8% was a model non-volatile core material (California Cooperative Creamery, Hughson, CA).

Microencapsulation

Spray-dried microcapsules containing AMF and core-free spray-dried powders were prepared from emulsions and wall solids solutions (Table 1). Core in wall emulsions and wall solutions were prepared as described by Young et al. (1993) and by Moreau and Rosenberg (1993, 1996). Spray drying was carried out at inlet- and outlet-air-temperature of 160°C and 80°C, respectively, using equipment and procedures described by Moreau and Rosenberg (1993, 1996, 1998). Particle size distribution in emulsions, microcapsule composition, and core retention during drying were determined as described by Young et al. (1993). Microstructural characteristics were investigated by SEM using methods previously described (Moreau and Rosenberg, 1993; Rosenberg and Young, 1993).

Porosity characteristics of microcapsules

Porosities of microcapsules and core-free spray-dried wall particles were investigated by gas displacement technique, using He and N_2 as permeating gases. Analyses were carried out using reported methodologies and equipment (Moreau and Rosenberg, 1998). A multipycnometer (Quantachrome Corporation, Boynton Beach, FL)

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Table 1—Composition of emulsions and mean core content in microcapsules. Wall solids concentration was 20% (w/w)

System	Lactose (%)[a]	AMF (%)[b]	AMF (%)[c]
WPI	—	—	—
WPI/L 3:1	25	—	—
WPI/L 1:1	50	—	—
WPI/L 1:3	75	—	—
WPI/L 3:1	25	75	74.02
WPI/L 1:1	50	75	74.43
WPI/L 1:3	75	75	74.28
WPI/L 1:1	50	25	24.75
WPI/L 1:1	50	50	49.32

[a]Lactose (% w/w) of wall solids.
[b]Anhydrous milkfat (% w/w of wall solids at the emulsion stage).
[c]Anhydrous milkfat (% w/w of nonfat solids in dry microcapsules).

connected to a high vacuum system was used for gas displacement analysis. The vacuum system consisted of an oil diffusion-pump (Model EO50/60, Edwards High Vacuum International, West Sussex, UK) equipped with a liquid nitrogen trap (#926300, Kontes, Vineland, NJ) and was backed with a mechanical pump (Maxima, Fisher Scientific, Pittsburgh, PA). Pirani Penning 1005 vacuum gauge equipped with a model PRM10 Pirani gauge head and a model CP25K Penning gauge head (Edwards High Vacuum International, West Sussex, UK) were used to monitor system pressure during degassing and gas-displacement procedures.

The volume of powder samples was determined using He and N_2 (separately) from the equilibrium pressure when a known volume of gas under pressure was allowed to flow from a volume-calibrated reservoir (Vr) into a volume-calibrated sample-cell. In all cases, Vr was calibrated at the temperature used for analyzing sample volume. Microcapsules (1-5 g) were placed in the sample-cell and degassed *in-situ* until pressure in the system remained constant below 2×10^{-3} Pa for a minimum of 4 h with a total degassing time of 8–12h (Moreau and Rosenberg, 1998). Following degassing, sample volume was determine using He as a displacing gas, then the system and sample were degassed again (as described), and sample volume was determined using N_2 as displacing gas. Volume was calculated from pressure changes during gas permeation. In all cases, V_r was pressurized to about 0.11 MPa. The equilibrium pressure (P_1) was read and recorded. Then, the measuring gas was introduced into the sample-cell from V_r, and pressure was recorded every 30s. The procedure for volume determination using a given measuring gas was repeated until three successive determinations agreed ±0.2%.

After volume determination with both gases, the weight of the sample microcapsules was determined. From the measured pressures, the reference volume (V_r) and volume of the sample-cell (V_c), the volume of the powder (V_x) was calculated.

$$V_x = V_c - V_r[(P_1/P_{2x}) - 1] \quad (1)$$

where: V_x = volume of the sample for a given time and gas (cm^3); V_c = calibrated volume of the sample-cell (cm^3); V_r = calibrated reference volume; P_1 was initial and P_{2x} was final pressure at time x.

Sample-volume determined at time x with He was designated $V_{H(x)}$; volume at time x determined with N_2 was designated $V_{N(x)}$. Densities of the sample in He and N_2 at time x ($\rho_{H(x)}$ and $\rho_{N(x)}$), respectively) were calculated using Eq (2) and (3), respectively:

$$\rho_{H(x)} = M/V_{H(x)} \quad (2)$$

$$\rho_{N(x)} = M/V_{N(x)} \quad (3)$$

where M=microcapsule weight (g); $V_{H(x)}$, $V_{N(x)}$=sample volume (at time x) in He and N_2, respectively.

Densities obtained with He and N_2 were compared to theoretical densities of the microcapsules calculated from density (at 25°C) and the weight fraction of each component of the microcapsule, as described by Moreau and Rosenberg (1998). Density of 1.400, 1.573, and 0.915 g/cm^3 were used for WPI, lactose, and AMF, respectively (Hayes, 1987; Walstra and Jenness, 1984).

At a given measuring time, the fraction of microcapsules pore-volume into which He could not penetrate was termed helium-restricted-volume and was calculated using the following equation:

$$Vp_{He(x)} = (1/\rho_{H(x)}) - (1/\rho_c) \quad (4)$$

where $\rho_{H(x)}$=density measured with helium at time x (g/cm^3); ρ_c=calculated density (g/cm^3); $Vp_{He(x)}$=helium-restricted-volume (cm^3/g).

Pore-volume of the microcapsules matrix that was accessible to He, but inaccessible to nitrogen was termed nitrogen-restricted-volume and was calculated as described by Moreau and Rosenberg (1998):

$$Vp_{N2(x)} = (1/\rho_{N(x)}) - (1/\rho_{H(x)}) \quad (5)$$

where $\rho_{N(x)}$ and $\rho_{H(x)}$ are density measured with N_2 and He at time x, respectively; $Vp_{N2(x)}$ is nitrogen-restricted volume at time x.

In all cases, replicate powders were analyzed at least in triplicate (n=6). Significance of results was tested at by ANOVA using the SigmaStat software (Jandel Scientific Software, San Rafael, CA). Significance of differences was defined at $P < 0.05$

RESULTS & DISCUSSION

IN ALL CASES, EMULSIONS USED TO PREPARE MICROCAPSULES exhibited unimodal particle size distribution. Mean particle size was not affected by emulsion composition and ranged from 0.31 to 0.34 μm. Core retention during microencapsulation ranged from 98.3 to 99.2% (Table 1) and was similar to reported data (Moreau and Rosenberg, 1993, 1996; Rosenberg and Young, 1993; Young et al., 1993; Rosenberg, 1997). Spherical microcapsules (20–60 μm in dia) exhibiting microstructural features (micrographs not shown) identical to those earlier reported by Moreau and Rosenberg (1993) were obtained. In all cases, microcapsules had a central void that accounted for 38.5–40% of capsule volume (calculated based on SEM results). In all cases, outer surface of microcapsules was free of visible pores or cracks and core material was distributed in the wall matrix in the form of small droplets (100–400 nm).

Results of gas displacement analyses (Fig. 1-3) indicated differences ($P<0.05$) in the way He and N_2 penetrated the microcapsules. In all cases, when He was the permeating gas, final pressure in the system was reached within 0.5–2.5 min and remained constant through-

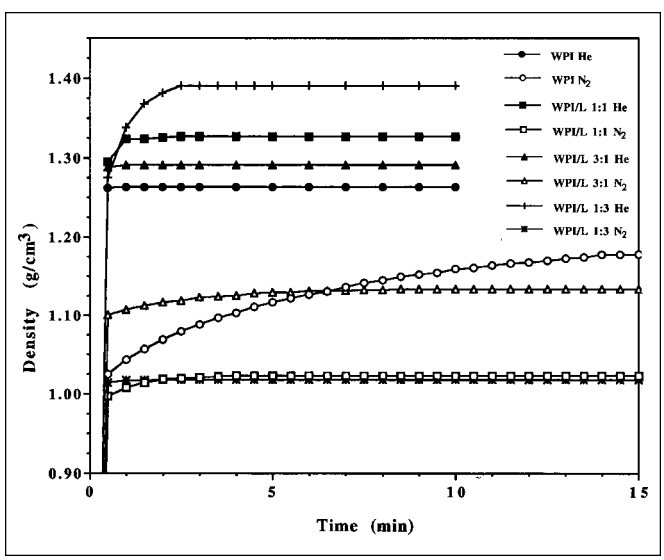

Fig. 1—Changes, with analysis time, in true and apparent density of core-free microcapsules with different wall composition. System code: WPI/L x:y - ratio of WPI-to-lactose. Wall solids concentration was 20%.

out the analysis time (15 min). When N_2 was the displacing gas, pressure exhibited a composition-dependent, continuous decrease over 15 min. Results indicated that He filled rapidly all the accessible volume while permeation of N_2 was hindered and thus significantly slower. Differences in gas permeation resulted in time-dependent differences between densities determined in He and N_2 (Fig. 1–3). Results of gas-displacement analyses could be attributed to a molecular-sieve type porosity, consisting of pores with molecular dimensions, that characterized both core-free and AMF-containing capsules (Rosenberg, 1985; Moreau and Rosenberg, 1998). Results thus indicated that sample volume and density determined with He should be referred to as "true volume" (V_t) and "true density" (ρ_t) and those determined with N_2 should be referred to as "apparent volume" (V_a) and "apparent density" (ρ_a), respectively (Moreau and Rosenberg, 1998). In order to avoid errors associated with temperature effects of pressure transducer (Quantachrome, 1991), equilibrium ρ_t and ρ_a were determined after 4 and 15 min, respectively.

Regardless of wall composition or core load, apparent density was lower than true density, throughout analysis, thus indicating that some pores were completely inaccessible to N_2 (Rosenberg, 1985; Moreau and Rosenberg, 1998). Wall composition and core load affected porosity of capsules. The overall smallest changes with time in apparent density were obtained with core-free systems consisting of 1:3 or 1:1 WPI/L (Fig. 1) thus suggesting the least porous wall matrix. This indicated that many pores in wall systems containing 50 or 75% lactose may have been completely impenetrable to N_2. Also, gas penetration into some parts of wall matrix containing $\geq$50% lactose may have been so slow that it could not be measured within the experimental time frame (Moreau and Rosenberg, 1998).

Equilibrium ρ_t value ranged between 1.086 and 1.390 g/cm^3 and was affected by core load and WPI-to-lactose ratio (Table 2). Permeation of He into core-free and 75% AMF-containing microcapsules was affected by the proportion of lactose included in the wall matrix (Fig. 1 and 2) and thus suggested the effects of lactose content on porosity of wall matrix. This could be attributed to the presence of lactose-based amorphous glassy phase in the wall systems of the spray-dried particles (Saltmarch and Labuza, 1980a, 1980b; Moreau and Rosenberg, 1993, 1996; Rosenberg, 1997). Effect of lactose on rate of He permeation could be attributed to increase in resistance of wall matrix to He penetration due to both complete sealing off of pores and to reduction of pore diameters. At a given WPI/L ratio, rate of He permeation was not affected by core load (Fig. 3).

Apparent density of the investigated microcapsule powders ranged from 0.820 to 1.110 g/cm^3 and from 0.900 to 1.134 g/cm^3 after 30s and 15 min, respectively, and was affected by both core load and wall composition (Fig. 1–3, Table 2). Rate of N_2 penetration into the microcapsules was affected by WPI/L ratio (Fig. 1 and 2). Comparing our results to those reported for WPI-based microcapsules (Moreau and Rosenberg, 1998) indicated that N_2 penetration into lactose containing wall matrices was appreciably slower ($>10\times$) than that into lactose-free WPI-based wall. These findings could be attributed to the influence of amorphous lactose glass on porosity. Rate of N_2 penetrated into AMF-containing microcapsules was faster (about $5\times$) than that into core-free systems (Fig. 1 and 3). This result could be explained through the reported effect of core load on microstructure (Rosenberg, 1985, 1988, Rosenberg and Lee, 1993; Rosenberg and Young, 1993). It has been reported that at a given wall solids concentration, thickness of wall matrices separating individual core domains was inversely related to core load (Rosenberg, 1985, 1988; Rosenberg and Young, 1993). Additionally, it has been established (Rosenberg and Lee, 1993) that WPI-based wall matrix consisted of a very dense layer of aggregated whey proteins adsorbed at the core/wall interfaces and a less dense matrix of proteins in the bulk of the wall system. At a given wall composition and core particle size-distribution, ratio between proportion of wall proteins associated with each of these phases was determined by core load. Results of our study thus suggested notable effects of core load on resistance of WPI/L wall systems to gas penetration, similar to that reported for microcapsules with WPI-only wall systems (Moreau and Rosenberg, 1998).

In all cases, ρ_t was lower than the theoretical density (Table 2) thus suggesting the presence of pores completely sealed off from He penetration. Differences between ρ_t and the theoretical density ranged from 10.1 to 11.8% and from 9.8 to 12.1% for core-free and AMF-containing microcapsules, respectively. The fraction of microcapsule volume that was inaccessible for He penetration, Vp_{He}, ranged from 0.066 to 0.082 cm^3/g and from 0.074 to 0.090 cm^3/g for core-free and AMF-containing microcapsules, respectively. Due to the very large central void that existed in all microcapsules, VpHe did not appear to be large enough to suggest exclusion of He from the central void. Results thus suggested that about 10–12% of the volume of the wall matrix consisted of pores that were sealed off from He penetration. These results could explain differences between the calculated and true densities that were evident in all cases. These results agreed with those reported by Moreau and Rosenberg (1998) for microcapsules

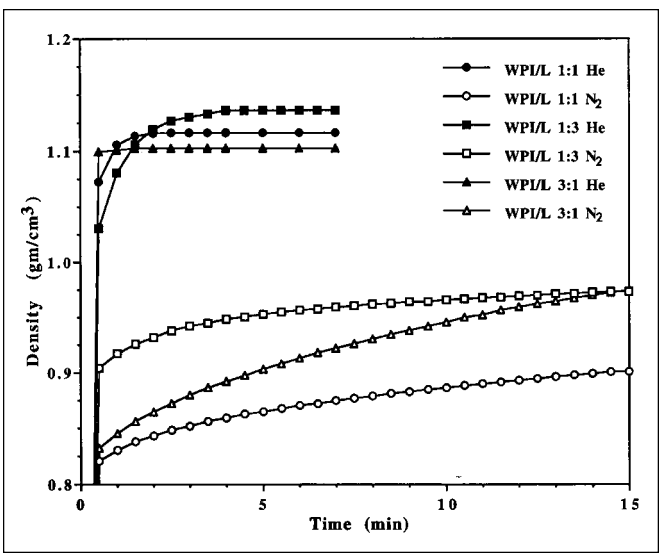

Fig. 2—Changes, with analysis time, in true and apparent density of 75% AMF-containing microcapsules with wall matrix consisting of different mixtures of WPI and Lactose. System code: WPI/L x:y - ratio of WPI-to-lactose. In all cases, total wall solids concentration was 20%.

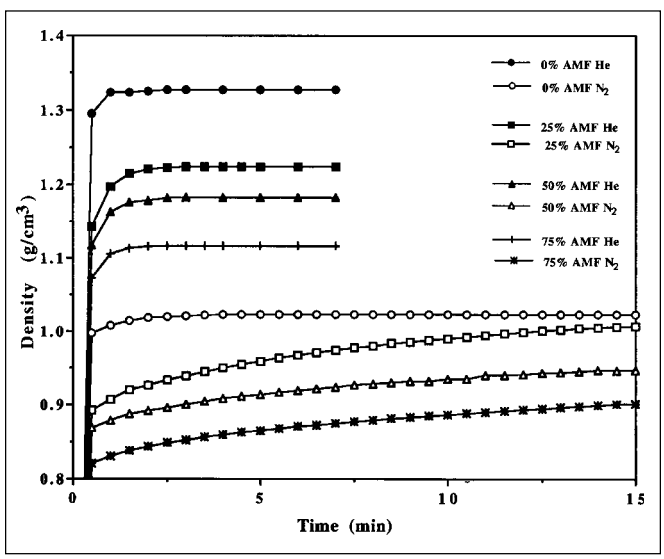

Fig. 3—Changes, with analysis time, in true and apparent density of AMF-containing microcapsules with wall matrix consisting of a 1:1 mixture of WPI and lactose. System code: % AMF - proportion (% w/w of dry wall solids) of AMF in emulsion before drying. In all cases, total wall solids concentration was 20%.

Table 2—True, apparent and calculated densities of AMF-containing and core-free microcapsules. Wall solids concentration was 20% (w/w)

System	AMF (%)[e]	Lactose(%)[f]	Density g/cm³		
			Theoretical	$_{t}$*	$_{a}$**
WPI	0	0	1.400	1.263[d]	1.178[a]
WPI/L 3:1	0	25	1.443	1.291[c]	1.134[b]
WPI/L 1:1	0	50	1.487	1.329[b]	1.023[c]
WPI/L 1:3	0	75	1.530	1.390[a]	1.018[c]
WPI/L 3:1	75	25	1.217	1.106[b]	0.975[b]
WPI/L 1:1	75	50	1.241	1.115[b]	0.900[c]
WPI/L 1:3	75	75	1.266	1.136[a]	0.973[b]
WPI/L 1:1	25	50	1.372	1.224[a]	0.993[a]
WPI/L 1:1	50	50	1.296	1.182[b]	0.947[b]

[a-d]Means within a column followed by different superscripts significantly different (P<0.05).
[e]Anhydrous milkfat (% w/w of wall solids at the emulsion stage).
[f]Lactose (% w/w) of wall solids.
*Equilibrium true density determined with He.
**Equilibrium apparent density determined with N_2

Table 3—Pore volume restricted to He (Vp_{He}) and N_2 penetration (Vp_{N2}) in Core-free and AMF-containing microcapsules. (In all cases, wall solids concentration was 20%)

System	AMF (%)[e]	Lactose (%)[f]	Vp_{He}	Vp_{N2} cm³/g	
			cm³/g	$Vp_{N1/2}$*	Vp_{N15}**
WPI	0	0	0.075[a]	0.187[b]	0.061[d]
WPI/L 3:1	0	25	0.082[a]	0.134[c]	0.108[c]
WPI/L 1:1	0	50	0.080[a]	0.250[a]	0.225[b]
WPI/L 1:3	0	75	0.066[a]	0.267[a]	0.263[a]
WPI/L 3:1	75	25	0.083[a]	0.297[ab]	0.121[c]
WPI/L 1:1	75	50	0.084[a]	0.321[a]	0.214[a]
WPI/L 1:3	75	75	0.090[a]	0.226[d]	0.148[b]
WPI/L 1:1	25	50	0.088[a]	0.309[a]	0.191[a]
WPI/L 1:1	50	50	0.074[a]	0.306[a]	0.211[a]

[a-d]Means within a column followed by different superscripts significantly different (P<0.05).
[e]Anhydrous milkfat (% w/w of wall solids at the emulsion stage).
[f]Lactose (% w/w) of wall solids.
*Determined after 30 s.
**Determined after 15 min.

with wall systems consisting of only WPI.

Equilibrium ρ_t ranged from 1.106 to 1.224 g/cm³ and from 1.263 to 1.390 g/cm³ for AMF-containing and core-free capsules, respectively. In all cases, trends observed for changes in equilibrium ρ_t agreed with those obtained with theoretical densities (Table 2). Equilibrium ρ_a ranged from 1.178 to 1.023 g/cm³ and from 0.993 to 0.900 g/cm³ for core-free and AMF-containing microcapsules, respectively. Among the powders differences in apparent density could not be explained based only on composition-related considerations. This therefore suggested effects of composition on particle porosity. Increasing the proportion of lactose in core-free particles from 0 to 75% decreased ρ_a by 15.7% rather than increasing it by 9.2%, as had been expected (Table 2).

For all AMF-containing microcapsules, differences ($P<0.05$) of 24.8–38.2% between theoretical and ρ_a were evident (Table 2). At a WPI/L ratio of 1:1, ρ_a was linearly and inversely related to core load ($r^2=1.00$). However, at a 75% AMF load, increasing lactose content from 25% to 50% (of wall solids) decreased the apparent density by 8.5% rather than increasing it by 19%. Further increase in lactose content to 75% resulted in ρ_a that was similar to that obtained at 25% lactose (Table 2). Results could be attributed to the influence of lactose content and core load on microstructure and hence porosity of microcapsules. These effects were reflected in differences observed in N_2 permeation into the different microcapsules (Fig. 1–3) and could be explained by differences in pore volume-fractions that were inaccessible to N_2 permeation.

Nitrogen-restricted-volume (Vp_{N2}) was calculated after 0.5 and 15 min of N_2 permeation and denoted $Vp_{N1/2}$ and Vp_{N15}, respectively. Differences between these parameters reflected effect of porosity profile on N_2 permeation (Rosenberg, 1985). Results (Table 3) indicated effects of composition on Vp_{N2}. For core-free particles, $Vp_{N1/2}$ increased with lactose content and accounted for 17.3, 33.2, and 37.1% of the V_t of particles prepared with wall systems having WPI/L ratio of 3:1, 1:1, and 1:3, respectively. Only a small decrease in Vp_{N2} with permeation time was observed for these systems and was inversely related to lactose content (Table 3). Vp_{N15} accounted for 13.9, 29.9, and 36.5% of the V_t of particles prepared with wall systems having WPI/L ratio of 3:1, 1:1, and 1:3, respectively. Results indicated that lactose-containing wall matrixes presented resistance to N_2 permeation. This in turn explained the relatively small changes with time in ρ_a observed for the lactose-containing systems (Fig. 1). This could also explain the found differences between theoretical and apparent densities. Moreau and Rosenberg (1998) reported that Vp_{N15} of core-free WPI-based particles accounted only for 7.7% of V_t of these particles. This value was significantly lower than that we obtained for the lactose-containing system. These differences highlighted the effecte of matrix composition on N_2 permeation (Fig. 1) and could be attributed to the presence of amorphous lactose-glass in the spray-dried particles (Saltmarch and Labuza, 1980a, b; Moreau and Rosenberg, 1993, 1996, Young et al., 1993, Rosenberg, 1997).

Results obtained with AMF-containing microcapsules (Table 3) indicated a combined effect of lactose content and core load on Vp_{N2}. $Vp_{N1/2}$ and Vp_{N15} of AMF-containing microcapsules accounted for 30.7-37.8% and 12.8-24.9 of V_t, respectively. $Vp_{N1/2}$ of microcapsules with 75% core load and wall systems containing 25 and 50% lactose was 2.2 and 1.28 larger than that of core-free particles with the same wall composition, respectively. These results could be attributed to the increase in resistance to gas permeation associated with the very dense WPI-based films adsorbed at the core droplet interfaces and the increase in tortuosity associated with presence of core particles embedded in the wall matrix (Rosenberg and Lee, 1993; Moreau and Rosenberg, 1998). However, the differences between VpN1/2 of AMF-containing and core-free systems decreased with lactose content and $Vp_{N1/2}$ of capsules with wall system containing 75% lactose was smaller (by 15.4%) than that of the corresponding core-free system. At a given wall composition, the amount of whey proteins available for matrix forming decreased with AMF content. This, and the similar core particle size-distribution suggested that the resistance to gas permeation was affected by both lactose content and proportion of WPI associated with the matrix bulk phase. It has been reported that decrease in protein content of wall bulk phase increased wall porosity (Moreau and Rosenberg, 1998). Results obtained with microcapsules with wall systems containing 75% lactose could thus be attributed to protein concentration in the bulk phase of the matrix that was low and its effect on $Vp_{N1/2}$ was greater than that of lactose content. Similar $Vp_{N1/2}$ was obtained for systems with wall systems consisting of WPI/L ratio of 1:1 and increasing core load (Table 3). At a given protein concentration, increasing core load decreased the proportion of protein associated with the matrix bulk phase. However, results suggested that at WPI/L ratio of 1:1 effect of amorphous lactose-glass phase on gas permeation was greater than that of protein content in the bulk phase of the matrix.

For all lactose-containing systems, Vp_{N15} was higher than that reported by Moreau and Rosenberg (1998) for systems consisting of only WPI or WPI and AMF. In all cases, changes in Vp_{N15} with composition exhibited trends similar to those observed for $Vp_{N1/2}$. Vp_{N15} accounted for 13.9–36.5% and 12.8 and 24.94% of the V_t of core-free and AMF-containing systems, respectively (Table 3). For AMF-containing systems, the highest resistance to N_2 permeation was exhibited at a WPI/L ratio of 1:1. Results suggested that at 75% core load and 25% lactose, effect of the core load on Vp_{N15} was greater than that of lactose, however, at 75% lactose the lactose effect was greater. These results agreed with the observed effect of lactose and core load on the microstructure of wall matrix. Vp_{N15} ranged from 80 to 98% and from 40.6 to 68.9% of $Vp_{N1/2}$ for core-free and AMF-containing systems, respectively. These results were higher that those reported for systems consisting of only WPI or WPI plus AMF where Vp_{N15} was only about 10% of $Vp_{N1/2}$. Results of our study regarding

Vp_{N2} thus clearly indicated that wall systems consisting of WPI and lactose exhibited resistance to N_2 permeation that was considerably higher than that presented by wall matrix consisting of only WPI.

CONCLUSION

MICROCAPSULES WITH WALL SYSTEMS CONSISTING OF WPI AND lactose exhibited molecular-sieve type porosity that was affected by wall composition and by core load. Results of our study could be used in designing microcapsule wall porosity needed for attaining desired mass transfer profile. Because of the similarity between molecular dimensions of nitrogen and oxygen, results with nitrogen could be used to predict oxygen permeation into microcapsules. This is important when functionality of wall systems in providing encapsulated core with protection against oxidation is considered.

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