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## **KINETICS OF WATER VAPOUR ADSORPTION AND CAKING OF WHEY POWDER**

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Key words: powdered whey, water vapour adsorption, caking

The study was aimed at investigating caking of sweet whey powder and whey demineralized as a result of water vapor adsorption in respect of their sorption capacity. The analysis of the course of kinetic curves demonstrated that sweet whey was characterised by a higher capacity and rate of water vapour adsorption as compared to the demineralized whey. During storage at relative humidity of RH>0.43 the powders were subject to caking once the material reached water activity ( $a_w$ ) of 0.3-0.4, yet the sticking of powder particles proceeded faster in the case of sweet whey.

# INTRODUCTION

Soluble substances present in food products are subject to phase transitions, the occurrence and rate of which are determined by the presence of water as well as other components. Dried food products are characterised by an amorphous form of soluble substances contained in them, which affects chemical and physical changes proceeding during their storage [Roos & Karel, 1991; Bhandari & Howes, 1999]. Crystallization of saccharides as a result of water sorption is linked with the problem of caking and sticking of powdered products [Aguilera et al., 1995; Wallack & King, 1988]. Dried dairy products are often the subject of investigations into the effect of lactose crystallization combined with water vapour sorption [Berlin et al., 1968; Saltmarch & Labuza, 1982; Linko et al., 1981; Lai & Schmidt, 1990; Chuy & Labuza, 1994; Domian & Lenart, 1999; Kim et al., 2002; Özkan & Walisinghe, 2002]. Description of changes proceeding requires the knowledge of both kinetics of water vapour adsorption as well as the state of sorption equilibrium [Boonyaia et al., 2004].

The research was aimed at investigating sorption capacity of powdered whey as well as its caking as a result of water vapour adsorption. The study involved also plotting isotherms of water vapour adsorption and analysis of the kinetics of water vapour adsorption and caking at three levels of relative humidity of the environment for two types of powdered whey.

#### MATERIAL AND METHODS

The experimental material were commercial sweet whey (SW) and demineralized whey (DW) produced by Lacma, Euroserum. Powdered wheys, containing 11-11.4% of protein, 1-1.5% of fat, 4% of water, differed in the content of lactose (SW – 72%, DW – 81%) and mineral salts (SW – 7%, DW – 1%).

The sorption capacity of the wheys was determined with the static (isotherms of water vapour adsorption) and dynamic method (kinetics of water vapour adsorption). Adsorption isotherms were analysed with the dessicator method at eleven levels of water activity (a<sub>w</sub>) from 0.0 to 0.903, within three months, at a temperature of 25±1°C. Adsorption kinetics was determined at three levels of relative humidity of the environment (RH 0.43, RH 0.65 and RH 0.81) at a temperature of  $25 \pm 1^{\circ}$ C within 50 h, using a stand that enabled keeping constant computer record of changes in sample mass in the medium with a given relative humidity (over a saturated salt solution) [Domian & Lenart, 1999]. Caking of whey (ca. 2-g samples) was carried out during storage in dessicators, at relative humidity of RH 0.43, RH 0.65 and RH 0.81, and a temperature of  $25 \pm 1^{\circ}$ C in the time span of up to 50 h. In a dessicator with a given RH, 26 samples were fixed, next after a specified period of time 2 samples were taken out each time. In those samples caking was evaluated visually after 10-fold rhythmical shaking of the flask. Evaluation of the degree of whey caking (O – a lack of caking;  $\Delta$  – partial caking;  $\Box$  – total caking) was accompanied by the measurement of water activity of the sample using a Hygroskop DT 1 apparatus (Rotronic AG, Switzerland) at a temperature of  $25 \pm 1^{\circ}$ C.

#### **RESULTS AND DISCUSSION**

Isotherms of water vapour adsorption for whey powders were characterised by a sigmoidal course consistent with type II isotherm according to BET classification (Figure 1). In the course of water vapour adsorption isotherm, at water activity range of 0.53-0.65 for sweet whey and water activity range of 0.43-0.53 for demineralized whey, there occurred discontinuity which was caused by the transition of amorphous lactose into the crystal-line state [Berlin *et al.*, 1968; Bronlund & Paterson, 2004].

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FIGURE 1. Isotherms of water vapour adsorption plotted for the examined whey powders: SW – sweet whey, DW – demineralized whey.

At a low water activity range ( $a_w < 0.4$ ), demineralized whey demonstrated slightly higher equilibrium water content as compared to the sweet whey; at that stage the main adsorptive component was lactose [Saltmarch & Labuza, 1982; Kim *et al.*, 2002]. At a higher relative humidity ( $a_w > 0.6$ ), the level of adsorbed water was affected, to a great extent, by the content of salts; the sweet whey was characterised by remarkably higher equilibrium water content as compared to the demineralized whey.

Adsorption of water vapour proceeded with a various rate, depending on the type of whey powder and relative humid-

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ity of the environment. In the case of powdered sweet whey (Figure 2a), at RH 0.43, the content of water was observed to increase with time, having reached the maximum value of 6.72 g/100 g d.m. after *ca*. 10 h; and then to slightly decrease to a value of 6.57 g/100 g d.m. after 50 h. In the kinetic curve of water vapour adsorption, at RH 0.65, there was observed a characteristic inflection once the whey powder reached water content of *ca*. 9.68 g/100 g d.m. in the 10<sup>th</sup> h, whereas the final water content stabilized at a level of 8.96 g/100 g d.m. At RH 0.81 there was observed a rapid increase of water content within 50 h up to the value of 15.99 g/100 g d.m.

Kinetic curves of demineralized whey were characterised by a similar course (Figure 2b). At RH 0.81 typical inflection appeared after *ca*. 4 h at a corresponding water content of 8.52 g/100 g d.m., at RH 0.65 – after *ca*. 3 h once water content reached the level of 7.28 g/100 g d.m., and at RH 0.43 – after *ca*. 10 h at water content of 6.72 g/100 g d.m. Relative state of equilibrium was reached after *ca*. 20 h of the process at RH 0.43 when water content accounted for *ca*. 6.2 g/100 g d.m; as well as at RH 0.65 and water content of 6.4 g/100 g d.m.; and at RH 0.81 and water content of 7.4 g/100 g d.m.

The greatest changes in the rate of water vapour adsorption, irrespective of RH value, occurred in the first 10 h of the process in the case of sweet whey and in the first 4 h in the case of demineralized whey (Figure 2). Adsorption rate was observed to decrease along with increasing water content of whey powders (Figure 3). In kinetic curves of water vapour adsorption there occurred a fragment of negative rate linked

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5 4 2 3 3 2 1 0 2 6 8 10 12 14 16 18 -1 <sup>4</sup> Water content (g/100 g d.m.) b 7 6 5 2 4 3 3 2 1 0 2 6 8 10 12 14 16 18 -1 4 Water content (g/100 g d.m.)

FIGURE 2. Kinetics of water vapour adsorption for sweet whey powder (a) and demineralized whey powder (b). Environment RH: 1 - 0.43, 2 - 0.65, 3 - 0.81.

FIGURE 3. Rate of water vapour adsorption (du/dt) as a function of water content (u) for sweet whey powder (a) and demineralized whey powder (b). Environment RH: 1 - 0.43, 2 - 0.65, 3 - 0.81.

	Sweet whey						Demineralized whey					
Time (h)	RH 0.43		RH 0.65		RH 0.81		RH 0.43		RH 0.65		RH 0.81	
	a <sub>w</sub>		a <sub>w</sub>		a <sub>w</sub>		a <sub>w</sub>		a <sub>w</sub>		a <sub>w</sub>	
0	0.22	0	0.22	0	0.22	0	0.23	0	0.23	0	0.23	0
0.5	0.24	0	0.26	0	0.35	$\diamond$	0.32	0	0.31	0	0.39	$\diamond$
1	0.28	0	0.30	$\diamond$	0.39	$\diamond$	0.33	0	0.33	0	0.43	$\diamond$
2	0.31	$\diamond$	0.36	$\diamond$	0.46		0.36	0	0.37	0	0.45	$\diamond$
3	0.32	$\diamond$	0.39	$\diamond$	0.53		0.37	0	0.39	$\diamond$	0.50	$\diamond$
4	0.34	$\diamond$	0.43		0.56		0.38	$\diamond$	0.40	$\diamond$	0.53	
5	0.38	$\diamond$	0.47		0.61		0.38	$\diamond$	0.42	$\diamond$	0.59	
6	0.41		0.50		0.63		0.39	$\diamond$	0.42		0.65	
7	0.41		0.52		0.65		0.40	$\diamond$	0.43		0.66	
8	0.42		0.52		0.64		0.42	$\diamond$	0.45		0.66	
9	0.42		0.53		0.65		0.42	$\diamond$	0.45		0.67	
10	0.43		0.55		0.64		0.41		0.46		0.69	
20	0.43		0.61		0.70		0.41		0.60		0.71	
50	0.41		0.60		0.74		0.40		0.61		0.72	

TABLE 1. Caking kinetics of powdered sweet and demineralized whey.

RH – relative humidity of environment;  $a_w$  – water activity of whey powder; degree of caking:  $\circ$  – a lack of caking.  $\diamond$  – partial agglomeration.  $\Box$  – total caking

with the phenomenon of desorption: for SW at RH 0.43 and 0.65 whereas for DW at RH 0.43, 0.65 and 0.81. The final rate of water vapour adsorption, irrespective of relative humidity of the environment and type of whey, tended to zero.

The occurrence of the inflexion effect of the kinetic curve of water vapour adsorption and the fragment of negative rate of the adsorption process was due to crystallization of amorphous lactose present in whey powders [Chuy & Labuza, 1994; Lai & Schmidt, 1990; Domian & Lenart, 1999]. The rate of lactose crystallization depended on water availability. A lack of inflexion of the kinetic curve of water vapour adsorption for demineralized whey at RH 0.81 was likely to result from the fact the water release during lactose crystallization was compensated for by water adsorption by other components, mainly mineral salts [Berlin *et al.*, 1968, Linko *et al.*, 1981].

The phenomenon of caking was observed both in the case of sweet and demineralized whey (Table 1). Whey powders were subject to caking at the three discussed levels of relative humidity, yet the process proceeded at a higher rate in the case of sweet whey. The caking was strictly lined with water activity of the powder [Chuy & Labuza, 1994; Özkan & Walisinghe, 2002]. For the sweet whey, that process occurred already at water activity range of  $a_w 0.3-0.4$ , whereas for demineralized whey caking occurred once the material had reached  $a_w$  of *ca*. 0.4.

# SUMMARY

Analyses of sorption capacity of whey powders demonstrated a significant effect of crystallization of amorphous lactose and content of mineral salts on the course of water vapour adsorption and whey powder caking. Sweet whey, not devoid of mineral compounds, demonstrated definitely higher capacity for water vapour adsorption and susceptibility to caking as compared to the demineralized whey.

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# KINETYKA ADSORPCJI PARY WODNEJ I ZBRYLANIA SERWATKI W PROSZKU

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Celem pracy było zbadanie zbrylania serwatki słodkiej i zdemineralizowanej w proszku w wyniku adsorpcji pary wodnej w porównaniu z ich właściwościami sorpcyjnymi. Analiza przebiegu krzywych kinetycznych wykazała, że serwatka słodka charakteryzowała się większą zdolnością i szybkością adsorpcji pary wodnej w porównaniu z serwatką zdemineralizowaną. Podczas przechowywania przy wilgotności powietrza 43% proszki ulegały zbryleniu po osiągnięciu przez materiał aw 0,3-0,4, przy czym zlepianie cząstek proszku następowało szybciej w przypadku serwatki słodkiej.