

Title	Influence of anti-caking agent on the water sorption isotherm and flow-ability properties of vacuum dried honey powder				
Authors	Nurhadi, Bambang;Roos, Yrjö H.				
Publication date	2017-04-20				
Original Citation	Nurhadi, B. and Roos, Y. H. (2017) 'Influence of anti-caking agent on the water sorption isotherm and flow-ability properties of vacuum dried honey powder', Journal of Food Engineering, 210, pp. 76-82. doi:10.1016/j.jfoodeng.2017.04.020				
Type of publication	Article (peer-reviewed)				
Link to publisher's version	10.1016/j.jfoodeng.2017.04.020				
Rights	© 2017 Elsevier Ltd. This manuscript version is made available under the CC-BY-NC-ND 4.0 license - http:// creativecommons.org/licenses/by-nc-nd/4.0/				
Download date	2025-07-08 14:26:43				
Item downloaded from	https://hdl.handle.net/10468/4052				



University College Cork, Ireland Coláiste na hOllscoile Corcaigh

Accepted Manuscript

Influence of Anti-Caking Agent on the Water Sorption Isotherm and Flow-ability Properties of Vacuum Dried Honey Powder

Bambang Nurhadi, Y.H. Roos

PII: S0260-8774(17)30167-X

DOI: 10.1016/j.jfoodeng.2017.04.020

Reference: JFOE 8856

To appear in: Journal of Food Engineering

Received Date: 06 February 2017

Revised Date: 14 April 2017

Accepted Date: 18 April 2017

journal of food engineering

Please cite this article as: Bambang Nurhadi, Y.H. Roos, Influence of Anti-Caking Agent on the Water Sorption Isotherm and Flow-ability Properties of Vacuum Dried Honey Powder, *Journal of Food Engineering* (2017), doi: 10.1016/j.jfoodeng.2017.04.020

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

Highlights (For Review)

- Effect of anti-caking agent addition to amorphous honey powder on its static and dynamic water sorption
- Effect of anti-caking agent addition to amorphous honey powder on its powder flow ability properties
- Effect of anti-caking agent addition on inhibiting recrystallization of amorphous sugar in vacuumdried honey powder

1	Influence of Anti-Caking Agent on the Water Sorption Isotherm and Flow-ability Properties of
2	Vacuum Dried Honey Powder
3	
4	Bambang Nurhadi ^{*)} and Y.H. Roos ^{**).}
5	^{*)} Faculty of Agro-Industrial Technology, Universitas Padjadjaran, Indonesia
6	**) School of Food Nutritional Sciences, University College Cork, Cork Ireland
7	
8	ABSTRACT
9	Honey powder is a hygroscopic powder due to its composition and structure. The addition of anti-
10	caking agent was aimed to increase the stability of honey powder. The present study aimed to
11	study the influence of anti-caking agent on the water sorption isotherm and flow-ability
12	properties of vacuum dried honey powder. Anti-caking agents, calcium silicate and calcium
13	stearate, were added in honey powder. The addition of anti-caking agent influenced water
14	sorption and flow-ability properties of honey powder. Calcium silicate addition seemed not to
15	affect water sorption isotherm properties of honey powder, however, calcium stearate showed
16	more inhibiting effect of recrystallization and collapse of structure of amorphous honey powder.
17	Addition of anti-caking agent to honey powder increase flow-ability of honey powder and calcium
18	stearate seemed to increase flow-ability better than calcium silicate.
19	
20	Key Words: Honey powder, anti-caking, calcium silicate, calcium stearate, flow-ability,
21	water sorption isotherm

23 1. Introduction

Honey powder is a hygroscopic powder due to its composition and structure. The two 24 main sugars found in honey solids, fructose, and glucose, have very low glass transition 25 temperatures making honey powder hygroscopic (Bhandari and Howes, 1999). Nurhadi and Roos 26 (2016a) reported the production of honey powder by using vacuum drying technique and drying 27 aid materials (filler), maltodextrin. Anhydrous honey powder glass transition temperature was 28 reported over 30 to 43 °C depending on drying aid material type and composition (Nurhadi and 29 Roos, 2016a). Critical water content of honey powder was also very low, less than 2 % (g 30 water/100 g sample) (Nurhadi and Roos, 2016a). Honey powder might be in purely amorphous 31 32 or partially amorphous structure depending on process used (Nurhadi and Roos, 2016b). When water is sorbed by honey powder during storage, water acts as a plasticizer, reduce glass 33 transition temperature of honey powder to lower than ambient temperature, results in 34 significant changes of macroscopic properties of honey powder, i.e. collapse which includes 35 phenomena such as stickiness, caking and shrinkage (Roos, 1995; Bhandari and Howes, 1999). 36 The rate of changes was dependent on the difference of ambient temperature to glass transition 37 temperature (T-T_g), the higher the difference was the faster was the rate at which the 38 39 macroscopic properties changed (Roos, 1995; Foster, Bronlund, Paterson, 2006; Roos and Drusch, 2015). The change would reduce quality of honey powder and the powder is becoming 40 difficult to handle and difficult to flow. 41

Method to keep honey powder stable during storage is by storing it at a temperature 42 below its glass transition temperature or below its critical water activity (Ca_w). Additionally, anti-43 caking agent could be used to maintain stability of sugar-rich powder such as honey powder 44 (Welti-Chanes et al., 2007). Anti-caking agent (or flow conditioner/free flowing agent) is the 45 46 ingredient added at low quantity to increase stability and maintain free flowing properties of host 47 material (Barbosa-Canovas et al., 2005). Anti-caking agent mechanism is different for amorphous and crystalline materials (Msagati, 2013). The mechanism of anti-caking agent might be by 48 competing for water with host material, acting as a water barrier, eliminating surface friction and 49 inhibiting crystal growth (Barbosa-Canovas et al., 2005 and Lipasek et al., 2012). Anti-caking agent 50 51 is classified as natural (such as kaolin, talc, and bentonite/silicate material) or synthetic when

52 manufactured from raw materials such as silicon dioxide (Msagati, 2013). Lipasek et al (2012) 53 reported the use of silicon dioxide, calcium silicate and calcium stearate up to 2 % as anti-caking 54 agent in host powder of crystalline material. The addition of anti-caking agent might affect water 55 sorption isotherm and flowing properties of host powder. The addition of anti-caking agent into 56 crystalline material did not change the shape of water sorption isotherm and some anti-caking 57 agent showed delaying deliquescence of crystalline material (Lipasek, Taylor and Mauer, 2011 58 and Lipasek et al., 2012).

Powder flow-ability is more complex than fluid (Xanthakis et al., 2015). Powder flow-59 ability is affected by material properties and stress history of material during processing (Koynov, 60 61 Glasser, Muzzio, 2015). Powder flow-ability is affected by internal factor such as particle size and distribution and external factor such as air humidity and temperature (Barbosa-Canovas et al., 62 2005). Powder flow-ability is one of powder bulk properties (Barbosa-Canovas et al., 2005). 63 Powder would flow if the load acting on the powder is bigger than the powder strength (or the 64 bulk structure of powder failed and start moving) (Barbosa-Canovas et al., 2005). Barbosa-65 Canovas et al (2005) considered powder flow-ability as a failure phenomenon. Two main 66 properties used to describe the flow-ability of powder are failure function and effective angle of 67 68 internal fraction. Failure function correlates major principal consolidation stress (MPS) and unconfined failure strength (UFS) to deduce the value of flow index. The correlation between 69 MPS and UFS is linear and the inverse slope of the best fit linear line equals to the flow index 70 (Barbosa-Canovas et al., 2005, Koynov, Glasser and Muzzio, 2015). The slope describes the 71 change of stress needed to induce the powder flow. The lower is the slope, the more free flowing 72 properties of the powder exist. The effective angle of internal friction is a property that describes 73 74 the inter particle interaction (Barbosa-Canovas et al., 2005) and powder with a low value of 75 effective angle is considered as a free flowing powder. The current study was aimed to investigate 76 the influence of anti-caking agents on water sorption and flow-ability properties of anhydrous 77 vacuum-dried honey powder.

78

79 2. Materials and Methods

80 2.1. Honey Powder

Honey was bought from local supermarket (Boyne valley Honey Brand) in Ireland. 81 Deionized water was used from KB scientific (Cork, Ireland) and maltodextrin DE 10 (M100) was 82 obtained from Grain processing corporation (IA, USA). Honey-maltodextrin solutions (total solid 83 43 % and ratio honey: maltodextrin = 6:4 and 4:6 (honey composition of 60 % and 40 % per 84 total solid, respectively)) were prepared on petri dishes with perforated covers for drying in 85 vacuum oven (WTB Binder, Germany) at temperature 60 °C for 3 hours. The mixture of honey, 86 maltodextrin and water is named as HMW. HMW 4:6 and HMW 6:4 referred to honey powder with 87 88 honey composition of 40 % and 60 % per total solid respectively. The dried samples were ground and stored in desiccator over P₂O₅ (Sigma-Aldrich) for further analysis 89

90

91

92 2.2. Mixing Honey Powder and Anti-Caking Agent

Anti-caking agents, calcium silicate and calcium stearate (Sigma-Aldrich), were used. Anti caking agents in Erlenmeyer flask and honey powder were added gradually based on geometric
 mixing principle to obtain an even distribution. Each addition of honey powder was followed by
 mixing with spatula.

97

98 2.3. Water Sorption Isotherm

99 **2.3.1. Static water sorption**

Static water sorption was prepared by storing sample of dried honey powder (+ 1 gram) in 100 101 desiccator over various saturated salt solutions. Seven different salt solutions were put in 102 desiccator to adjust the relative humidity (RH). The salt solutions used were LiCl, CH₃COOK, MgCl₂, K₂CO₃, Mg (NO₃)₂, NaNO₂, and NaCl (Sigma Chemical Co., St. Louise, MO. U.S.A) to adjust 103 RH value of 11 %, 23 %, 33 %, 43 %, 52 %, 65 % and 75 %, respectively (Roos, 1995). The weight 104 change of samples was monitored regularly (every day) for three weeks or until steady weight 105 was reached. The water content of samples from each desiccator were determined by drying in 106 vacuum oven at 100 °C for 6 hours. The data of water content of honey powder and their 107

108 corresponding water activity was used to make water sorption isotherm (WSI) curve. The 109 Guggenheim-Anderson-deBoer (GAB) equation was used as a model of WSI.

110
$$X = \frac{X_m C K a_w}{(1 - K a_w)(1 + (C - 1) K a_w)}$$
(1)

111

112 Where,

113 X = water content (g water/ g dry solid)

114 a_w = water activity

- 115 X_m = monolayer water content
- 116 X_m, C, and K are constants

117

118 **2.3.2.** Dynamic water sorption (Dynamic Dew point Isotherm/DDI)

119 Dried sample (about 500 mg) was used in DDI (Decagon Devices, Inc. Pulman WA, USA) 120 measurements. The adsorption direction of dynamic water sorption was set up. The measurement were conditioned as follows, temperature of 25 °C, air flow of 300 ml/min and aw 121 122 range starting from 0.1 to 0.85. In DDI adsorption direction, the wet air pass over the dried 123 sample and the sample would adsorb moisture from the air. If the initial water activity of dried sample higher than 0.1 a_w, the dry air would pass over the sample until its water activity less than 124 125 0.1, then adsorption direction would be started. The data of sample weight change and its corresponding water activity were recorded at certain interval time. The initial water content of 126 dried sample was determined by reducing water activity below 0.1 a_w and the predicted water 127 128 content then processed by DDI software (SorpTract Software, Decagon, Device Inc., Pulman WA, 129 USA). The data collection continued until the final set water activity was reached. The data of 130 sample weight changes were converted into data of sample water content. Finally the data of 131 sample water content and its corresponding water activity would be used to draw dynamic water 132 sorption curve.

133

134 2.4. Tapped Density

Honey powder sample was put in 25 ml measuring cylinder until fully covered the volume stated.The measuring cylinder with honey powder inside then was put in tapped density meter (J.

 $H_R = \frac{\rho_{\infty}}{\rho_o}$

Engelsmann AG, Germany) and tapping was done for 100 times. The end volume was then
 recorded. Hausner Ratio (H_R) was calculated as followed (Barbosa-Cannovas et al., 2005):

139

140

141 Where :

142 ho_{∞} = tapped density (bulk density of powder after tapping)

143 $\rho_o =$ initial loose bulk density

144

145 2.5. Powder Flow ability

Flow ability of honey powder was measured by using Powder Flow tester (Brookfield, USA). 146 147 Sample of honey powder was put in trough whose annular shape and flattened with inner catch tray. The trough has volume 38 cc. the initial weight of trough and the weight after sample 148 149 addition were recorded. The vane lid was then moved down to cover the trough. The vane lid had diameter of 12.7 cm and curved profile. Standard flow test was set to run the experiment. 150 151 The trough then rotated while powder consolidated/compressed. Five consolidation stresses 152 were used. Five consolidation stresses used were 0.795, 1.607, 3.246, 6.559 and 13.252 kPa. As an example at consolidation stress of 0.795 kPa, the 3 stresses set points used were 0.795 KPa 153 and the other two stresses lower than 0.795 kPa (0.265 and 0.536 kPa) (Fig. 1). The trough rotated 154 until steady flow reached and then the shear stress and normal stress were recorded. Data of 155 normal stress and shear stress were recorded for each consolidation stress. From Fig. 1, major 156 principal consolidation stress (MPS) and unconfined failure strength (UFS) were determined for 157 each consolidation stress with the use of powder flow pro. V1.2 Build 19 (Brookfield, USA). The 158 159 best fit line of normal stress and shear stress was called as yield locus and Mohr's semi-circle was 160 used to determine MPS and UFS (Barbosa-Cannovas et al., 2005 and Koynov, Glasser and Muzzio, 2015). UFS was determined with the use of Mohr's semi-circle which passing through origin point 161 and tangent to the yield locus while MPS was determined with the use of Mohr's semi-circle 162 163 which passing through pre shear consolidation stress (0.597 KPa) and tangent to the yield locus. MPS and UFS represented the condition for the critical state and critical failure respectively 164

(Koynov, Glasser dan Muzzio, 2015). Effective yield locus was determined based on the line which passing through origin and tangent to bigger Mohr's semi-circle and the angle between x-axes and effective yield locus was called as effective angle of internal friction (Barbosa-Cannovas et al., 2005). The data between MPS and UFS for every consolidation stress was then correlated to determine flow factor index. Flow factor index was the inverse of slope of best fit linear line between MPS and UFS.

- 171 Based on the value of flow factor index (ff), flow ability of a powder can be classified as (Barbosa-
- 172 Cannovas et al., 2005).
- 173 ff < 1 non flowing
- 174 1 < ff <2 very cohesive
- 175 2 < ff < 4 cohesive
- 176 4 < ff < 10 easy flowing
- 177 ff > 10 free flowing
- 178
- 179
- 180 Fig 1. Yield Locus, effective yield locus, and Mohr's stress semi-circle from a sample of honey
- 181 powder (HMW 4:6) flow ability measurement by powder flow tester at 0.795 kPa consolidation
- 182 stress.
- 183

184 **3. Results and Discussion**

185 3.1. Water Sorption Isotherm

186 Water sorption isotherm of honey powder was done by using static/conventional and dynamic method (DDI method). Even though the data from dynamic water sorption could not be 187 compared with data obtained from conventional method in calculating water sorption model 188 (such as GAB and BET model) (Schmidt and Lee, 2012), the DDI data could be used to study the 189 properties changes related to their corresponding water activities (Romani et al., 2016) such as 190 191 critical water activity determination (Yuan, Carter and Schmidt, 2011; Carter and Schmidt, 2012), 192 deliquescent point (Ghorab et al., 2014) and amorphous content (Nurhadi and Roos, 2016b). 193 From Fig. 2, it can be seen that addition of anti-caking agent (2%) to host powder of honey powder did not change shape of WSI and the WSI were classified as type II of Brunaeur water 194 195 sorption isotherm classification (Labuza and Altunakar, 2007). Nurhadi and Roos (2016a) also reported that honey powder had type II of Brunaeur water sorption isotherm classification. The 196 result agreed with those reported by Lipasek, Taylor and Mauer (2011) that the anti-caking agent 197 did not change the WSI shape of deliquescent material. As shown in Fig. 2, anti-caking agent 198 199 addition to honey powder also did not change its corresponding monolayer water content.

- 200
- 201

Fig. 2. Static water sorption isotherm of honey powder and honey powder with addition of anticaking agent and GAB parameter for each treatment.

204

Honey powder (maltodextrin ratio of 60 % per total solid) with calcium stearate addition 205 (2%) had higher water sorption than other two treatments of honey powder over water activity 206 (a_w) range of 0.44-0.76 (Fig. 2). As shown by Fig. 3a, the significant increase of water sorption of 207 208 honey powder occurred above a critical water activity (around 0.35 a_w). The Ca_w of honey powder was higher than the Ca_w of honey powder (determined based on static water sorption) reported 209 by Nurhadi and Roos (2016a). The different result was caused by the different water sorption 210 method and the Ca_w obtained from dynamic WSI determination usually higher than the Ca_w value 211 based on static WSI and DSC (differential scanning calorimeter) glass transition measurement 212 (Carter and Schmidt, 2012). The critical value obtained by DDI method is much affected by the 213

214 flow rate of air over the sample during the measurement. The higher is the air flow rate, the 215 higher is the critical water activity obtained (Yuan, Carter and Schmidt, 2011). The Ca_w from 216 normal method (DSC and WSI method) might be equal to the Ca_w obtained from DDI method by extrapolating the Ca_w at zero flow rate (Yuan, Carter and Schmidt, 2011). From Fig. 3.a. It could 217 218 be seen that anti-caking addition seemed not to change the critical water activity of honey powder. The critical water activity was correlated with the significant increase of water sorption 219 220 due to transition from glassy structure to rubbery structure of amorphous component (Yuan, 221 Carter and Schmidt, 2011; Carter and Schmidt, 2012). After critical water activity surpassed, the macroscopic properties such as crystallization of amorphous components and caking increased 222 223 significantly (Nurhadi and Roos, 2016b). As shown in Fig. 3a, there were differences in water sorption after 0.5 aw between honey powder with calcium stearate and other two honey powders 224 (honey powder and honey powder with calcium silicate). From DDI WSI result, the "equilibrium 225 water content" for both calcium stearate and calcium silicate corresponded to 0.1-0.8 aw, were 226 of 0.2 % - 0.5% and 1.8 % - 6.0 %, respectively. The low water sorption of both calcium stearate 227 and calcium silicate were also reported by Lipasek et al. (2012). 228

The clearer differences of water sorption behavior between honey powders are shown in 229 230 Fig. 3b. Nurhadi and Roos (2016b) used first derivative of DDI WSI curve to show crystallization of amorphous components during water sorption which appeared as a peak and the area of peak 231 correlated to the extent of crystallization occurred. It seemed that the crystallization of 232 amorphous components of honey powder occurred, however, the inhibition of crystallization 233 234 was apparent in honey powder with addition of calcium stearate. Honey powder without anticaking agent and honey powder with calcium silicate had more complete peak than the honey 235 236 powder with calcium stearate (Fig. 3b).

- 237
- 238
- 239 240

241

b.

a.

Fig. 3. Dynamic water sorption of honey powder and honey powder with the addition of anticaking agent (a), first derivative curve of dynamic water sorption of honey powder and honey powder with the addition of anti-caking agent (b).

245

From static WSI experiment, the collapsed structure of honey powder appeared in honey 246 powder without anti-caking agent and honey powder with calcium silicate starting at 0.44 a_w 247 while collapse did not occur in honey powder with calcium stearate (Fig. 4). However at higher 248 a_w, honey powder with Calcium stearate addition also showed stickiness and lump phenomena 249 $(0.65 \text{ and } 0.75 \text{ a}_{w})$ and need tapping the vial several time to move out the powder from the vial. 250 Calcium stearate act as a barrier and due to its hydrophobic properties to repel water from 251 252 ambient air to the host particle (honey powder) that might show some inhibition effect of crystallization of amorphous sugar (maintaining structural integrity) and collapse. Lipasek et al. 253 (2012) explained the ability of calcium stearate as moisture barrier between particles that might 254 interrupt bridging mechanism as an initial step of collapse occurred. Murrieta et al. (2011) stated 255 the collapsed structure of milk powder was triggered by recrystallization of amorphous lactose 256 in milk powder. The collapse phenomena occurred when Ca_w surpassed and caused physical and 257 chemical changes such as non-enzymatic browning reaction (Roos, 1995, Roos and Drusch, 2015). 258 259 Thus it could be shown that the addition of anti-caking agent (Calcium stearate) into honey powder gave some effect of inhibition of crystallization and collapse of amorphous structure of 260 honey powder during water sorption experiment. That explained the phenomena occurring in 261 static WSI of honey powder (Fig. 2) where honey powder with calcium stearate had higher water 262 sorption than the other two honey powders due to recrystallization inhibition of amorphous 263 sugar in honey powder. The addition of anti-caking agent, calcium silicate and calcium stearate 264 at 1% in honey powder showed the same result as those with 2% (data not shown). The 265 266 disadvantages of calcium stearate addition are related to its taste and solubility.

267

268

Fig. 4. Honey powder and honey powder with addition of anti-caking agent after reaching
 equilibrium at various water activity, from top to bottom correspond to honey powder, honey
 powder + 2 % calcium stearate and honey powder + 2 % calcium silicate, respectively.

272

- 273
- 274

275 **3.2. Flow-ability**

In order to work optimally as anti-caking agent, there should be interaction or surface 276 277 affinity between anti-caking agent and host powder (Barbosa-Canovas et al., 2005). The interaction is indicated by the increase of bulk density of host powder after addition of an anti-278 caking agent (Barbosa-Canovas et al., 2005). As shown in Table 1, the addition of anti-caking 279 agent (calcium stearate and calcium silicate) increased bulk density from 0.59 to 0.64-0.66 280 (g/cm³). The increase of bulk density showed the high surface affinity between anti-caking and 281 282 host particle and if it is not, anti-caking agent could fill the void between host particles powder and no increase of bulk density may occur (Barbosa-Canovas et al., 2005). 283

284

Table 1. Parameters of honey powder obtained from powder flow tester and tapped densitytester.

287

Flow-ability of honey powder with higher maltodextrin ratio was better than the lower 288 ratio. As given in Table 1 and Fig. 5, the flow-ability index of honey powder increased from 289 290 cohesive to easy flowing powder when the ratio of maltodextrin to honey increased from 40 % to 60 % (solid ratio). Honey powder with lower maltodextrin ratio had lower glass transition 291 temperature (Nurhadi and Roos, 2016a) and tended to be more hygroscopic. The DDI critical 292 water activity of honey powder increased (from around 0.3 a_w to 0.35 a_w) when the maltodextrin 293 ratio to honey increased from 40 % to 60 % (per total solid) (Fig. 6). Stoklosa et al. (2012) stated 294 295 that flow-ability of powder was influenced significantly by external conditions such as air relative humidity of storage. When water was adsorbed by honey powder, water acted as a plasticizer 296 297 and reduced glass transition temperature of honey powder to below ambient temperature (Roos 298 and Drusch, 2015) and honey powder became more sticky and difficult to flow.

- 299
- 300
- 301

Fig. 5. The flow-ability characteristic of honey powder and honey powder with addition of anti-caking agent.

- 304
- 305

Fig. 6. Dynamic water sorption of honey powder at different ratio of maltodextrin DE 10

Instead of increasing the ratio of maltodextrin as a drying aid material, anti-caking agent 308 might be used to improve the flow-ability of honey powder. As can be seen in Table 1 and Fig. 5, 309 310 the addition of anti-caking agent 2 % (both calcium stearate and calcium silicate) could increase the flow-ability properties of honey powder from an easy flowing to a free flowing powder. 311 312 Density properties were also used as an indication of powder flow-ability (Barbosa-Canovas et al., 2005 and Xanthakis, Ommen and Ahrne, 2015). Relative decrease of bulk density after 313 compaction or tapping of powder was an indicator that the powder had good flow-ability 314 properties (Barbosa-Canovas et al., 2005). From Table 1, based on the value of Hausner ratio (H_R), 315 the honey powders (excluding honey powder with lower maltodextrin ratio, HMW 6:4) were 316 classified as a medium flowing powder while HMW 6:4 was classified as a difficult flowing 317 powder. The increasing trend of bulk density value after compaction in powder flow-ability tester 318 319 of honey powder with anti-caking agent were smaller than that of honey powder with anti-caking 320 agent and the result complied with the flow index results. However, the contrary result based on density measurement was found in honey powder with different maltodextrin ratio. The contrary 321 result of flow-ability measurement based on density properties was also reported by Xanthakis, 322 Ommen, and Ahrne (2015). 323

Flow-ability of powder was determined as a failure function in term of flow index as 324 explained in Barbosa-Canovas et al (2005). In addition to flow index, the effective angel of 325 326 internal friction can also be used to determine the flow-ability properties of honey powder. The 327 lower the value of effective angle of internal friction, the more free flowing properties the 328 powder has (Barbosa-Canovas et al., 2005). As shown in Fig. 7, honey powder with calcium stearate had the lowest effective angle of internal friction followed by both honey powder 329 330 without anti-caking agent and honey powder with calcium silicate and honey powder with lower 331 maltodextrin ratio (HMW 6:4). The effective angle of internal friction is a property that described

332	inter-particle interaction of powder. The lower value of the angle indicated a smoother surface
333	interaction between particles in the powder. Compared to calcium silicate, calcium stearate has
334	bigger molecular size and had ability to elongate and narrow better than calcium silicate to give
335	protection of the surface more extensively. Lipasek et al (2012) also reported calcium stearate as
336	the best anti-caking agent in reducing effective angle of internal friction of crystalline sucrose.
337	
338	

339	Fig. 7. Effective angle of internal friction of honey powder and honey	powd	ler with	addition	of
340	anti-caking agent.				

343 **4. Conclusion**

344 Anti-caking agent calcium silicate and calcium stearate were added into honey powder. 345 The addition of anti-caking agent influenced water sorption and flow-ability properties of honey powder. Calcium silicate addition seemed not to affect water sorption isotherm properties of 346 honey powder, however calcium stearate showed some inhibiting effect of recrystallization and 347 collapse of structure of amorphous components of honey powder. Addition of anti-caking agent 348 to honey powder increase flow-ability of honey powder and calcium stearate seemed to increase 349 flow-ability better than calcium silicate. Addition of anti-caking agent increased flow-ability of 350 351 honey powder from an easy flowing to a free flowing powder and calcium stearate showed 352 significant reduction of internal friction between particles of honey powder that contributed the powder becoming more free flowing. 353

354

355					
356	References				
357	Bhandari, B. R., & Howes, T. (1999). Implication of glass transition for the drying and stability of				
358	dried foods. Journal of Food Engineering, 40(1), 71–79. <u>http://doi.org/10.1016/S0260-</u>				
359	<u>8774(99)00039-4</u> .				
360	Bhandari and Roos. 2012. Food material science and engineering : an overview in Food Material				
361	Science and Engineering. Bhandari, B., and Roos, Y.H. (eds). Blackwell Publishing ltd. West				
362	Sussex				
363	Barbosa-muriet Canovas, G.V., E.O. Rivas, P. Juliano and H. Yan. 2005. Food Powders. Physical				
364	Properties, Processing, and Functionality. Kluwer Academic/Plenum Publisher. New York.				
365	Carter, B.P., and S.J. Schmidt. 2012. Developments in glass transition determination in foods				
366	using moisture sorption isotherms. Food Chemistry 132: 1693-1698.				
367	Chmielewska, H.R. 2004. Honey. in Chemical and Fuctional Properties of Food Saccharides.				
368	Tomasik, P. (ed.). CRC Press. Boca Raton.				
369	Foster, K. D., Bronlund, J. E., & Paterson, a. H. J. (T). (2006). Glass transition related cohesion of				
370	amorphous sugar powders. Journal of Food Engineering, 77(4), 997–1006.				
371	Ghorab, M.K., K. Marrs, L.S. Taylor and L.J. Mauer. 2014. Water-solid interaction between				
372	amorphous maltodextrin and crystalline sodium chloride. Food Chemistry 144: 26-35.				
373	Koynov, S., Glasser, B., & Muzzio, F. (2015). Comparison of three rotational shear cell testers:				
374	Powder flowability and bulk density. Powder Technology, 283, 103–112.				
375	http://doi.org/10.1016/j.powtec.2015.04.027.				
376	Labuza, T.P., and B. Altunakar. 2007. Water Activity Prediction and Moisture Sorption Isothermal. In Water				
377	Activity in Foods. Fundamentals and Applications. Cannovas, G.V.B., A.F. Fontana Jr., S.J. Schmidt, T.P.				
378	Labuza (eds). IFT Press. Iowa.				
379	Lipasek, R. A., Taylor, L. S., & Mauer, L. J. (2011). Effects of Anticaking Agents and Relative				
380	Humidity on the Physical and Chemical Stability of Powdered Vitamin C. Journal of Food				
381	Science, 76(7), 1062–1074. http://doi.org/10.1111/j.1750-3841.2011.02333.x.				
382	Lipasek, R. a., Ortiz, J. C., Taylor, L. S., & Mauer, L. J. (2012). Effects of anticaking agents and				
383	storage conditions on the moisture sorption, caking, and flowability of deliquescent				
384	ingredients. Food Research International, 45(1), 369–380.				

385 http://doi.org/10.1016/j.foodres.2011.10.037.

- Murrieta-Pazos, I., Gaiani, C., Galet, L., Cuq, B., Desobry, S., & Scher, J. (2011). Comparative
 study of particle structure evolution during water sorption: Skim and whole milk powders.
- 388 Colloids and Surfaces B: Biointerfaces, 87(1), 1–10.
- 389 http://doi.org/10.1016/j.colsurfb.2011.05.001
- 390 Msagati, T.A. 2013. Chemistry of food additives and preservatives. Wiley-Blackwell. West Sussex.
- Nurhadi, B., & Roos, Y. H. 2016a. Water Sorption and Water Plasticization Behavior of Vacuum
 Dried Honey. *International Journal of Food Properties* 19: 1370-1380.
- Nurhadi, B., & Roos, Y.H. 2016b. Dynamic Water Sorption for the Study of Amorphous Content
 of vacuum dried honey powder. Journal of Powder Technology : (under review).
- 395 Romani, S., P. Rocculi, S. Tappi, and M.D. Rosa. 2015. Moisture Adsorption Behavior of Biscuit
- during Storage Investigated by Using a New Dyanamic Dew Point Method. Food Chemistry
 195: 97-103
- 398 Roos, Y.H. 1995. Phase transition in foods. Academic Press. San Diego, C.A.
- Roos, Y.H. and S. Drusch. 2016. Phase transition in foods second edition. Academic Press. Oxford.
- 400 Schmidt, S. J., & Lee, J. W. (2012). Comparison between Water Vapor Sorption Isotherms
- 401 Obtained Using the New Dynamic Dewpoint Isotherm Method and Those Obtained Using
- 402 the Standard Saturated Salt Slurry Method. International Journal of Food Properties, 15(1-
- 403 2), 236–248. http://doi.org/Doi 10.1080/10942911003778014
- Stoklosa, A. M., Lipasek, R. A., Taylor, L. S., & Mauer, L. J. (2012). Effects of storage conditions,
 formulation, and particle size on moisture sorption and flowability of powders: A study of
 deliquescent ingredient blends. *Food Research International*, 49(2), 783–791.
 http://doi.org/10.1016/j.foodres.2012.09.034
- Welti-Chanes, J., E. Perez, J.A.G. Beltran, S.M. Alzamora and F.V. Balderas. 2007. Application of
 water activity management in the food industry. In Water Activity in Foods. Fundamentals
 and Applications. Canovas, G.V.B., A.F. Fontana Jr., S.J. Schmidt, T.P. Labuza (eds). IFT
 Press. Iowa.
- 412Xanthakis, E., Ruud van Ommen, J., & Ahrné, L. (2015). Flowability characterization of413nanopowders.PowderTechnology,286,156–163.

414 http://doi.org/10.1016/j.powtec.2015.08.015.

- Yuan, X., B.P. Carter, and S.J. Schmidt. 2011. Determining the critical relative humidity at which
 the glassy to rubbery transition occurs in polydextrose using an automatic water vapor
 sorption instruments. Food Science 76: 78-89.



Fig 1. Yield Locus, effective yield locus, and Mohr's stress semi-circle from a sample of honey powder (HMW 4:6) flow ability measurement by powder flow tester at 0.795 kPa consolidation stress.



Fig. 2. Static water sorption isotherm of honey powder and honey powder with addition of anticaking agent and GAB parameter for each treatment.



Fig. 3. Dynamic water sorption of honey powder and honey powder with the addition of anticaking agent (a), first derivative curve of dynamic water sorption of honey powder and honey powder with the addition of anti-caking agent (b).



Fig. 4. Honey powder and honey powder with addition of anti-caking agent after reaching equilibrium at various water activity, from top to bottom correspond to honey powder, honey powder + 2 % calcium stearate and honey powder + 2 % calcium silicate, respectively



□ HMW 6:4 ■ HMW 4:6 ▲ HMW 4:6-Ca Stea 2% ● HMW 4:6-Ca Sil 2%

Fig. 5. The flow-ability characteristic of honey powder and honey powder with addition of anticaking agent.

-



Fig. 6. Dynamic water sorption of honey powder at different ratio of maltodextrin DE 10



Fig. 7. Effective angle of internal friction of honey powder and honey powder with addition of anti-caking agent.

	Powder flow-ability tester			Tapped Density			
		Density (g/cm ³)			(g/cm ³)		
Treatment	Flow index, ff	Initial	After Consolidation 6.5 kPa	Relative Increase	ρ _o	ρ _t	H _R
HMW 6:4	3.04 <u>+</u>	0.49 <u>+</u>		1.49 <u>+</u>	0.58 <u>+</u>	0.75 <u>+</u>	1.3 <u>+</u>
	0.26	0.03	0.72 <u>+</u> 0.02	0.03	0.01	0.00	0.03
HMW 4:6	7.64 <u>+</u>	0.59 <u>+</u>		1.57 <u>+</u>	0.67 <u>+</u>	0.81 <u>+</u>	1.21 <u>+</u>
	0.43	0.01	0.91 <u>+</u> 0.03	0.06	0.01	0.01	0.00
HMW 4:6-Ca	11.91 <u>+</u>	0.66 <u>+</u>	1 05 1 0 12	1.49 <u>+</u>	0.70 <u>+</u>	0.85 <u>+</u>	1.22 <u>+</u>
Stea 2 %	2.55	0.03	1.05 ± 0.12	0.11	0.01	0.02	0.00
HMW 4:6-Ca	13.44 <u>+</u>	0.64 <u>+</u>	0.00 + 0.02	1.39 <u>+</u>	0.70 <u>+</u>	0.81 <u>+</u>	1.16 <u>+</u>
Sil 2 %	0.38	0.01	0.90 ± 0.02	0.03	0.00	0.00	0.00

Table 1. Parameters of honey powder obtained from powder flow tester and tapped density tester.