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# 1 Powders flowability assessment in granular compaction: what about the 2 consistency of Hausner ratio?

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## 6 7 8 1. Introduction 9

10 Among the wide range of granular materials, products manufactured in powder form are  
11 present in many industries such as chemical, pharmaceutical, cosmetic, food and polymers  
12 industries [1]. A powder is a set of heterogeneous particles dispersed in a continuous gas  
13 phase.

14 Powders processing involves different unit operations such as aeration, drying, milling,  
15 compaction, handling, storage and conveying [2]. In order to insure the feasibility of the  
16 process chain, good powders flowability is required.

17 Nevertheless, powders flow behavior is complex in nature because is not an inherent property  
18 of the material. In fact, it depends on: the intrinsic and extrinsic properties of the grains such  
19 as size, shape, surface rugosity, porosity and crystal chemistry [3]; the bulk powder properties  
20 such as size distribution, bulk density, interaction forces between particles; the equipment  
21 design; the processing conditions such as stress levels; the processing environment such as  
22 temperature and humidity.

23 The complexity of the link between local particles interactions and their global mechanical  
24 behavior has undermined flowability understanding, remaining essentially empirical.

25 Among the greatest challenges in powder technology, the main trend in industrial applications  
26 is to develop an effective device allowing to predict powder flowability by reproducing the  
27 mechanical conditions encountered during processing.

28 In order to allow a better understanding of the powder/process relation, flow properties of  
29 powders can be categorized in groups corresponding to the stress levels applied to the  
30 powder: over packed bed, free surface and aerated conditions [4]. Different methods can be  
31 used in order to reproduce these stress levels:

- 32 • Packed bed conditions: consolidation tester (Jenike's cell, FT4, RST-XS...), uniaxial  
33 compression (Instron 4505, Stylcam® 100R rotary press, Gerristen test, Johanson  
34 test);
- 35 • Free surface conditions: angle repose (Granuheap®, BEP2® ...), vibratory  
36 compaction (Densitap®, Hosokawa®, Granupack®), flow rate through apertures

37 (Gardco®, Granuflow®, BEP2®, Flodex®...), rotating drum (Aero-Flow®,  
38 Granudrum®);

39 • Aerated conditions: rotating drum-fluidization test (Granudrum®, The  
40 Revolution®...), fluidization bed (FT4- aeration test, mini-Glatt).

41 Different parameters or index can be obtained from these testers such as angle of repose  
42 (AoR), Hausner ratio (HR), Carr's index (i.e., compressibility) (CI), basic flow energy (BFE),  
43 flowability index ( $ff_c$ ), flow rate index (FRI), fluidization quality (FQ) and aeration index (AI).  
44 Each one of these allows to "estimate/quantify" flow behavior under different mechanical  
45 situations.

46 Hausner ratio and Carr's index are two closely related empirically derived methods that allow  
47 to assess the flow behavior from bulk densities. Quite popular in industry and academia,  
48 because of the simplicity and the rapidity of the measurement, both compression ratios were  
49 obtained under quite different reflections. Carr suggested in 1965: "*it is obvious that*  
50 *compressibility is a very important flow characteristic... the more compressible a material is,*  
51 *the less flowable it will be*" [5]. Introduced by Hausner in 1967 while studying copper  
52 powders, HR was described as "*an indicator of the friction condition between powder*  
53 *particles*" [6].

54  
55 In this study, *powder flowability under free surface conditions is determined by using HR.*  
56 The flow properties of powders, according to HR, were assessed using three vibratory  
57 compaction devices: the well-known DensiTap®, GranuPack® which is an automated version  
58 of DensiTap® from Granutools® and a homemade Vibratory device initially developed for  
59 granular rheology, allowing to control the dynamic parameters of the motion [7].

60 The main difference between these devices is the way of shaking, in DensiTap® and  
61 GranuPack® the vibration is generated by free-falls performances, commonly denoted as taps.  
62 The tapping amplitude, or intensity, corresponds to the free-fall height. In the case of our  
63 Vibratory device, the vibration is harmonically driven by an electromagnetic [shaker excited](#)  
64 [with a sinusoidal signal](#).

65  
66 The aim of this work is to get a deeper understanding of HR [by confronting these compaction](#)  
67 [tests, pointing out their relevance considering the physical sense of the measurement. To do](#)  
68 [so, it is suitable to approach flowability from a physical vibrated granular materials point of](#)  
69 [view. Granular compaction takes place when a pile of grains is submitted to a series of taps](#)  
70 [and the packing fraction of the pile, defined as the ratio of the volume occupied by all the](#)

71 grains divided by the volume of the assembly, slowly increases. The compaction and  
72 flowability of granular materials are still a serious challenge for physicist, and so, most efforts  
73 have been made to relate thermodynamic approaches for understanding granular media. Most  
74 of these studies are carried out with ideal or model granular materials such as glass beads.  
75 Among these works, the ones on compaction dynamics agreed that the granular media  
76 subjected to shocks, or taps, slowly compact to a stationary state [8, 9, 10]. Ribière et al. 2007  
77 showed that this stationary state is independent of the initial state of the powder, slightly or  
78 not dependent of the vibration frequency but is strongly dependent of the vibration amplitude  
79 [11]. Ludewig et al. (2008), proposed an energetic dimensionless parameter for investigating  
80 the physical properties of dense granular systems which seems to capture the entire dynamics  
81 of compaction [12]. The energy injected by a tap or sinusoidal signal seems to be a key  
82 parameter for describing the compaction dynamics. From this observation, also validated in  
83 this work, we propose, that, the energy injected to the granular media is also a key parameter  
84 for describing powders flowability through HR assessment.

85 Here we experimentally study granular materials submitted to vibration under free surface  
86 conditions. The mainly focus is on the properties of the stationary state for determining HR as  
87 a flowability index and discuss the validity of such configurations in function of the vibration  
88 energy supplied to the granular system. For this, when possible, the vibration will be  
89 described as a function of the energy injected to the system. The aim is to develop an  
90 energetical point of view for describing flowability by the use of HR.

91  
92 In order to illustrate the consistency of the results, a variety of non-ideal, or more  
93 complicated, granular materials notably different from glass beads in terms of nature, shape,  
94 polydispersity, roughness, even cohesion behavior, have been selected to cover a large range  
95 of applications from pharmaceutical to food industries.

96

97

## 98 **2. Materials and methods**

99

### 100 *2.1. Materials*

101 For the current study, nine powders samples have been selected according to their size  
102 distribution, particles shape and their flow behavior observed with naked eye. Among them,  
103 two grades of lactose (L): Tablettose 70 and Retalac; two grades of microcrystalline cellulose  
104 (MCC): Avicel ph 102 and Avicel ph 105, and five commercial food powders (FP) denoted

105 Food A, Food B, Food C, Food D and Food E respectively. The commercial name of the food  
106 powders is kept confidential.

- 107 • Tablettose 70 is the trade name for  $\alpha$ -lactose monohydrate produced by MEGGLE  
108 (Germany), which particles are characterized by a narrow size distribution. According  
109 to the technical brochure, Tablettose 70 is especially designed for direct-compression  
110 and exhibits a good flowability.
- 111 • Retalac is lactose powder produced by MEGGLE (Germany), composed by equal  
112 parts of hypromellose polymer and  $\alpha$  –lactose monohydrate. Retalac powders are  
113 characterized by excellent flow and compaction properties.
- 114 • The two grades of Avicel, ph 102 and ph 105, produced by FMC Biopolymer, are high  
115 purity microcrystalline cellulose particles. Both powders differ only by their particles  
116 size distribution. Avicel ph grades are well known in the pharmaceutical industry for  
117 direct compression tableting applications.
- 118 • Food A and C are vegetal powders, Food B is a mineral powder, Food D is a [dietary](#)  
119 fiber, and Food E is a protein. Food A, Food B and Food C are characterized by  
120 having a poor flowability observed with naked eye. All food powders particles differ  
121 from each other in size and shape.

122

123 It should be noted that, [since water vapor can introduce cohesion between the particles and](#)  
124 [change their frictional properties](#), at the beginning of the experiments, all powders samples  
125 were conditioned at a relative humidity (RH) of 30%. During analysis, samples were keep at  
126 laboratory environmental conditions, between 30 and 40 % RH.

127

## 128 *2.2. Powders physical characterization techniques*

129

### 130 *2.2.1. Particle size analysis*

131

132 Particle size distributions were measured by laser diffraction in liquid media with a  
133 Mastersizer 2000 from Malvern Instruments. Depending on the nature of the particles, water  
134 or ethanol were used in order to improve their dispersion avoiding dissolution. All samples  
135 were analyzed in triplicate to ensure reproducibility.

### 136 *2.2.2. Particle morphology analysis*

137 Particles morphology analyses were assessed with a Scanning Electron Microscopy (Joel JSM  
138 T330A), and a field emission gun operating between 5kV to 15 kV. Samples were placed onto

139 carbon tapes and coated with gold during 5 min using an Ion SPUTTER JFC-1100 Jeol under  
140 argon gas purge.

### 141 2.2.3. True particles density analysis

142 Powder true density analysis was performed in an Helium pycnometer Accupyc 1330 from  
143 Micromeritics. All data measurements are the average of ten measurements obtained from  
144 one powder sample.

145

### 146 2.3. Hausner ratio determination

147 Hausner ratio can be defined as the ratio of the tapped bulk density to the loose bulk density  
148 as follows:

149

$$150 \quad HR_{\infty} = \frac{\rho_{\infty}}{\rho_0} \quad (1)$$

151

152 where  $\rho_0$  is the aerated density [obtained after freely pouring the powder in to the vessel](#) and  
153  $\rho_{\infty}$  [is the asymptotic constant density obtained during tapping until no further volumes](#)  
154 [changes occurs, also described as the stationary state](#) [6, 13, 14].

155 A more practical equation widely used to evaluate flow properties is given by volume changes  
156 in a graduated cylinder after certain number of taps,  $N$ :

157

$$158 \quad HR = \frac{\rho_N}{\rho_0} = \frac{V_0}{V_N} = \frac{C_N}{C_0} \quad (2)$$

159

160 where  $\rho_0$  and  $\rho_N$  are the aerated and tapped density,  $V_0$  and  $V_N$  are the initial and the tapped  
161 volume,  $C_0$  and  $C_N$  are the initial and tapped compactness ( $C_0 = 1 - \varepsilon$ , with  $\varepsilon$  the porosity of  
162 [the aerated bed](#)).

163

164 HR according to Carr's classification, as indicator of the flowability characteristics of the  
165 powder, has been reported elsewhere [5, 15].

166

167 It should be noted that in the following the accuracy of HR is calculated using instrumental  
168 and random errors analysis as follows:

$$169 \quad E_T = \sqrt{E_R^2 + E_I^2} \quad (3)$$

170 
$$E_I = \frac{V_0}{V_N} \sqrt{\left(\frac{\Delta V_0}{V_N}\right)^2 + \left(\frac{\Delta V_N}{V_N}\right)^2} \quad (4)$$

171 
$$E_R = \sqrt{\frac{1}{n-1} \sum_{k=1}^n (HR_k - \overline{HR})^2} \quad \text{with} \quad \overline{HR} = \frac{1}{n} \sum_{k=1}^n HR_k \quad (5)$$

172

173 where  $E_T$ ,  $E_R$  and  $E_I$  are respectively the total, random and instrumental errors,  $n$  is the  
 174 number of repetitions and  $\overline{HR}$  the [mean value](#) of Hausner ratio.

175

176 *2.4. Flowability testers: vibratory compaction under free surface conditions*

177

178 *2.4.1. DensiTap®*

179 This device allows determining aerated and tapped densities by mechanically tapping a  
 180 graduated glass cylinder. A rotating cam provides the tapping action by raising the cylinder  
 181 platform through a fixed distance of  $A = 3$  mm at a frequency of 4.16 Hz.

182 [As described in the European Pharmacopeia](#), about 150 mL of powder are freely poured by  
 183 using a funnel into the vessel allowing to determine aerated density while tapped density is  
 184 determined systematically after 500 taps [15]. This number of taps, the most used in scientific  
 185 literature, is proposed as the number of taps suitable to reach the steady state.

186

187 *2.4.2. Granupack®*

188 Developed by Granutools®, this device automatically measures [the position of a hollow](#)  
 189 [cylinder placed on the top of the powder column. From this distance, the height and the](#)  
 190 [volume of the powder bed are computed](#) allowing to determine the evolution of the packing  
 191 fraction as function of the number of taps.

192 The metal cylindrical enclosure containing the powder sample performs free falls with an  
 193 amplitude of 1 or 3 mm while the tap frequency can be tuned from 0,1 to 2 Hz.

194

195 The Granupack® software allows to plot the compaction kinetic curve by monitoring the  
 196 intermediate states. Other dynamical parameters can be extrapolated from the compaction  
 197 curves, using for example Bideau's model [9]:

198

199 
$$C(N) = C_\infty - (C_\infty - C_0) \exp\left[-\left(\frac{N}{\tau}\right)^\beta\right] \quad (6)$$

200

201 where  $C_\infty$  is the extrapolated compactness when  $N$  tends to infinity,  $\tau$  is the characteristic tap  
202 number related to the compaction dynamics and  $\beta$  is a stretching exponent.

203

204 The kinetic parameters  $\tau$  and  $\beta$  have been used in others studies to differentiate powders  
205 having similar HR, with  $\beta$  related to powder cohesiveness [9, 16]. In this work, we are only  
206 interested on the determination of  $C_\infty$ . The aim is to compare  $HR$  from  $HR_\infty$  values.

207

208 As suggested by Granutools®, to determine HR, a powder sample of 35 mL is poured in the  
209 metallic cylinder. Free fall events are carried out at a fixed frequency of 1 Hz and the two  
210 tapping amplitudes, 1 and 3 mm, allowed by the device have been tested. The compaction  
211 curves are obtained from 0 to 500 taps.

212

### 213 2.4.3. Vibratory system

214

215 This homemade device is a particle damper where a cylindrical vessel in borosilicate glass is  
216 harmonically driven by an electromagnetic shaker (Fig.1). In this case, each tap is defined by  
217 a sinusoidal motion of the container. The motion of the system is given as:

218

$$219 \quad x(t) = A_0 \cdot \sin(2\pi f \cdot t) \quad (7)$$

220

221 Where  $A_0$  and  $f$  correspond to the amplitude and frequency of the signal and characterize the  
222 motion.

223

224 The dynamic of the system is experimentally obtained by an accelerometer glued to the  
225 vessel. Thus, the amplitude of the motion,  $A_0$ , can be determined by using the root mean  
226 square acceleration,  $a_{RMS}$ , as follows:

227

$$228 \quad A_0 = \frac{a_{RMS} \cdot \sqrt{2}}{(2\pi f)^2} \quad (8)$$

229

230 Finally, the mechanical vibration energy,  $E_v$ , injected to the powder bed can be calculated:

231



232 
$$E_v = \frac{1}{2} m (2\pi f)^2 A_0^2 \quad (9)$$

233

234 where  $m$  is the powder mass.

235 This device allows to measure what we define as  $HR_\infty$ . Indeed the final compaction state is  
236 determined after no further volume changes occur, also known as the stationary state ( $\rho_\infty$ ).

237 As the stationary state is slightly or not dependent on the vibration frequency but is strongly  
238 dependent on the vibration amplitude [11] a frequency of 30 Hz was chosen because it  
239 allowed the wider amplitude scanning, from 10 to 110 m.s<sup>-2</sup>.

240 It should be noted that most studies in granular compaction when working with harmonically  
241 driven vibrations use a frequency of 30 Hz [8, 10, 12] or even 60 or 90 Hz [11] but discretize  
242 the movement by adding regular intervals of 1 s. The basic idea of using discrete “shakes” is  
243 to allow sufficient time between excitations so that all motion in the column from one shake  
244 ceased before the next starts. In our case, when working with continuous signals, we assume,  
245 as the typical time scale of rearrangements of rigid particles, also called the *confinement time*,  
246 is highly inferior ( $T_{p,max} \sim 0,2$  ms) to one period (33 ms), that each successive vertical shake  
247 are separated by a time delay that allows the total relaxation of the granular assembly [17].  
248 This can also be validated by the fact that each HR value is obtained at one steady-state  
249 density, and so the granular system reaches a stationary state during continuous vibration.

250 In terms of tapped density, at a vibration frequency of 30 Hz, each  $HR_\infty$  value was obtained  
251 after at least 5000 periods (comparable to 5000 taps).

252 The experimental procedure is the following: 30 g of powder are systematically poured into  
253 the container to determine  $\rho_0$ . Once the frequency of the excitation signal is fixed at 30 Hz the  
254 amplitude of the signal is progressively increased, resulting in an increase of the acceleration  
255 (measured  $a_{RMS}$ ). It should be noted that each amplitude scanning experiment takes around  
256 45 min per sample.

257

258 In order to allow a better comparison between the devices, their characteristics and operating  
259 conditions are summarized in Table 1.

260

261  
262  
263  
264  
265

**Table 1**  
Devices characteristics

Characteristics / Device	Densitap®	Granupack®	Vibratory system
Type of vibration	shock wave	shock wave	sinusoidal
Vessel dimensions [mm] (diameter / height)	34.7 / 335	26 / 155	26 / 300
Quantity of powder [mL]	150	35 *	~ 80 (30 g)
Vessel material	borosilicate glass	stainless steel	borosilicate glass
Amplitude of vibration [mm]	1 *	1 ou 3 *	0 to 3
Frequency of vibration [Hz]	4.16 *	1	30 **
HR determination	500 taps*	500 taps*	Stationary state

266 \* Fixed or proposed by the supplier.  
267 \*\* Chosen to obtain a maximal amplitude scanning.  
268

269

### 3. Results and discussion

270  
271

#### 3.1. Physical characterization results

272  
273

274 Powders physical properties are reported in Table 2. Particles shape are displayed in Fig .2  
275 (Food Powders), Fig. 3 (MCC powders) and Fig. 4 (Lactose powders). All SEM micrographs  
276 are presented with a bar scale of 20 µm, 50 µm and 100 µm.

277

278 **Table 2**  
279 Physical properties of the samples  
280

Powder	Particle size distribution ± 1 [µm]				True density ± 0.01 [g/mL]	Shape [-]
	d <sub>10</sub>	d <sub>50</sub>	d <sub>90</sub>	Span[-]		
Food A	7	15	38	2.2	1.46	Isometric rough in agglomerate state
Avicel ph 105	4	18	40	2.0	1.56	Rod Isometric rough
Food B	4	29	115	3.8	2.28	Isometric smooth
Food C	29	104	155	1.9	1.47	Irregular

Avicel ph 102	29	104	248	2.0	1.57	Rod Isometric rough
Food D	65	120	221	1.3	1.45	Agglomerates pop corn like
Retalac	89	203	408	1.6	1.42	Agglomeration of amorphous-like particles
Food E	123	267	472	1.3	1.23	Isometric rough
Tablettose 70	118	204	380	1.3	1.54	Individual agglomerates with a rough structured surface

281

282

283 SEM micrographs of Food samples are shown in Fig. 2. Food A and Food D are formed by  
 284 agglomerates of amorphous-like particles and pop-corn like particles respectively. Food B is  
 285 composed by oval shape particles were smaller particles get stuck on the surface of the largest  
 286 ones. Food C is composed by bigger irregular particles with rough surfaces. Food E is  
 287 composed by the most irregular particles, closer zooms exhibit a porous surface.

288

289 Pharmaceutical excipients, Avicel ph 102 and Avicel ph 105, are composed by rough rod  
 290 particles (Fig. 3). Avicel ph 105 particles are smaller than Avicel ph 102.

291

292 Lactose samples are shown in Fig. 4. Tablettose 70 powder exhibit better-defined particles  
 293 with a rough structured surface. Retalac particles are the agglomeration of crystalline alpha  
 294 lactose and hypromellose fibers resulting in amorphous-like particles.

295

296 Particles size distribution are presented in Fig. 5 (Food powders), Fig. 6 (MCC powders) and  
 297 Fig. 7 (Lactose powders). As depicted from Table 2 and Fig. 5, Food A particles are the  
 298 **thinnest** ones, Food B present the **widest** span, Food C present a bimodal distribution, Food D  
 299 show a narrow size distribution while Food E is composed of the biggest particles.

300

301 Particle size distribution of MCC powders show that particles of Avicel ph105 are smaller  
 302 than of Avicel ph 102 particles (Figure 6) while the two lactose samples have a similar  
 303 particle size distribution (Figure 7).

304

305

### 306 *3.2.Flowability results*

307

#### 308 *3.2.1. Assessment of powder flowability with DensiTap® device*

309

310 In order to study the sensitivity of Hausner ratio to the initial state of the powder samples, two  
 311 batches were selected for their quite different flow behavior: Tablettose 70 (good flowability)  
 312 and Food A (bad flowability). For each powder, the initial and final volumes were measured  
 313 by testing different filling methods: gently, rapid, with or without funnel, changing the  
 314 operator, etc. All data, presented in Table 3, are the average of 15 measurements on 15  
 315 different samples from the same batch.

316

317 **Table 3**

318 Accuracy of the measurements for 15 and 3 repetitions for Tablettose 70 and Food A.

Powder	HR (15 repetitions)	HR (3 repetitions)
Tablettose 70	1.15 ± 0.01	1.14 ± 0.01
Food A	1.26 ± 0.02	1.25 ± 0.01

319

320

321 The good repeatability of the results, according to the reading of initial and final volume after  
 322 tapping, showed an easily reproducible initial state (Table 3). It should be noted that we do  
 323 not know if this aerated state obtained after pouring the powder is the loosest packing that  
 324 could be achieved, but proved to be a rather constant value so that repeated measurements,  
 325 starting from the same initial conditions, could be made. These results are in disagreement to  
 326 those obtained by several authors, suggesting that HR values are strongly affected by the  
 327 highly irreproducible initial pouring of the powder [4, 16].

328 Moreover, as depicted from Table 3, the use of 3 measurements seems to be enough to obtain  
 329 an acceptable accuracy to determine HR. Thus, in the following, all HR values calculations  
 330 are obtained as the average of 3 measurements.

331 The results obtained for all powders are gathered in Table 4 according to increasing HR.

332

333 **Table 4**

334 Powders flowability analysis derived from Hausner ratio using a DensiTap® device with  
 335  $f_t = 4.16$  Hz,  $A = 3$  mm and  $N = 500$  taps.

Powder	$d_{32}$ (μm)	$d_{43}$ (μm)	HR ( $A = 3$ mm)	Flow behavior
Tablettose 70	184	244	1.14 ± 0.01	Good flow
Food D	102	133	1.15 ± 0.02	Good flow
Food E	178	283	1.21 ± 0.01	Fair flow
Food C	24	81	1.22 ± 0.02	Fair flow

Food A	8	26	$1.25 \pm 0.01$	Fair flow
Retalac	159	229	$1.26 \pm 0.02$	Passable flow
Food B	10	49	$1.28 \pm 0.03$	Passable flow
Avicel ph 102	40	126	$1.31 \pm 0.04$	Passable flow
Avicel ph 105	8	20	$1.44 \pm 0.04$	Poor flow

336

337

338 Several authors have shown that powders ability to flow increases as particles size increases  
 339 [18, 19]. Generally [this is](#) related to an increase of the gravitational forces which become  
 340 preponderant compared to interparticle forces due to an increase of particles individual mass.  
 341 In our case, HR analysis with  $f_t = 4.16$  Hz and  $A = 3$  mm, non-clear conclusions can be  
 342 drawn by size distribution analysis. [For example, lactose powders, Tablettose 70 and Retalac,](#)  
 343 [composed by the biggest particles, exhibit quite a different flow behavior mainly attributed to](#)  
 344 [the highly heterogenous shape of Retalac powders. Nevertheless, this statement based in](#)  
 345 [particles morphology and size doesn't explain flowability differences in the case of food](#)  
 346 [powders.](#)

347

### 348 3.2.2. *Flowability assessment with Granupack® device*

349

350 The evolution of powders compactness as a function of the number of taps for an amplitude of  
 351 1 and 3 mm are presented, respectively, in linear and logarithmic scales in Fig. 8, Fig. 9,  
 352 Fig.10 and Fig.11 ([Error bars are shown for each experimental point](#)).

353

354 As can be seen from Fig. 8 and Fig.10, the compactness increases with the number of taps.  
 355 The monitoring of compaction as function of number of tap allows to visualize more easily if  
 356 the steady state is reached. But, the representation in logarithmic scale (Fig. 9 and Fig. 11)  
 357 shows that the steady state is not always established as suggested by the representation in  
 358 linear scale (case of Avicel ph 102, Retalac and Food D).

359

360 In Tables 5 and 6 are gathered the HR values obtained with both tapping amplitudes, 1 and  
 361 3 mm respectively. According to the index classification from Table 5, it can be depicted that  
 362 most powders exhibit a [fair](#) ability to flow. In difference, from Table 6, [most of the samples,](#)  
 363 [exhibit another classification.](#) Thus, [HR assessment](#) seems to be strongly dependent on the  
 364 free-fall amplitude.

365

366

367 **Table 5**368 Flow behavior under Granupack® deduced by Hausner ratio ( $f_t = 1$  Hz,  $A = 3$  mm,  $N = 500$ )

369

Powder	$d_{32}$ ( $\mu\text{m}$ )	$d_{43}$ ( $\mu\text{m}$ )	HR ( $A = 3$ mm)	Flow behavior
Food E	178	283	$1.15 \pm 0.02$	Good flow
Food C	24	81	$1.22 \pm 0.01$	Fair flow
Retalac	159	229	$1.24 \pm 0.01$	Fair flow
Avicel ph 102	40	126	$1.24 \pm 0.01$	Fair flow
Tablettose 70	184	244	$1.24 \pm 0.03$	Fair flow
Food D	102	133	$1.25 \pm 0.01$	Fair flow
Food B	10	49	$1.28 \pm 0.02$	Passable flow
Food A	8	26	$1.31 \pm 0.01$	Passable flow
Avicel ph 105	8	20	$1.42 \pm 0.01$	Poor flow

370

371

372

373 **Table 6**374 Flow behavior under Granupack® deduced by Hausner ratio ( $f_t = 1$  Hz,  $A = 1$  mm,  $N = 500$ ).

375

Powder	$d_{32}$ ( $\mu\text{m}$ )	$d_{43}$ ( $\mu\text{m}$ )	HR ( $A = 1$ mm)	Flow behavior
Food E	178	283	$1.13 \pm 0.01$	Good flow
Food C	24	81	$1.16 \pm 0.01$	Good flow
Retalac	159	229	$1.16 \pm 0.01$	Good flow
Food A	8	26	$1.17 \pm 0.03$	Good flow
Food D	102	133	$1.19 \pm 0.02$	Fair flow
Food B	10	49	$1.20 \pm 0.03$	Fair flow
Avicel ph 102	40	126	$1.21 \pm 0.01$	Fair flow
Tablettose 70	184	244	$1.25 \pm 0.01$	Fair flow
Avicel ph 105	8	20	$1.29 \pm 0.02$	Passable flow

376

377

378 To obtain  $C_\infty$  values and so  $HR_\infty$ , all compaction curves were fitted using Eq. (6). For all

379 samples, Bideau's model was found to be in good agreement with the kinetics of compaction

380 ( $R^2 \approx 0.99$ ) as shown in Fig. 12.

381

382

383

384 Fig. 12 and Fig. 13 show the compaction curve and the compaction model, for Avicel ph 102,  
385 in linear and logarithmic scales respectively. The logarithmic representation of the data,  
386 Fig.13, shows a slight deviation from the model for  $N < 10$ . The compaction model chosen  
387 for this study has been established using glass beads [9] thus, the discrepancy observed can be  
388 explained by the use, in our case, of more complex samples.

389 The comparison between  $HR$  and  $HR_{\infty}$  values is presented in Fig. 14. For most powders,  
390 statistically similar HR values were obtained from  $C_{N=500}$  and  $C_{\infty}$  for a fixed amplitude,  
391 showing that the use of 500 taps to evaluate HR, is a quite good choice as a standard  
392 procedure and can explain why this number of taps is the most used in scientific literature  
393 [13]. Only for Retalac samples  $HR$  and  $HR_{\infty}$ , obtained with  $A = 1$  mm, are statistically  
394 different. This exception can be explained by being too far from the steady state with  $N = 500$   
395 taps, as can be depicted from Fig. 11.

396 Fig. 14 allows to better apprehend at which point HR is strongly dependent on the tapping  
397 amplitude. Indeed, most of the samples present a different flowability classification when  
398 changing the free-fall amplitude. Only Food E and Tablettose 70 powders possess statistically  
399 similar HR values.

400

401

### 402 3.2.3. Flowability assessment with a vibratory device

403

404 The evolution of powders compactness as function of the vibration amplitude at a fixed  
405 frequency of 30 Hz are presented in Fig. 15. The reorganization of all powders studied seems  
406 to be similar with a variation of compactness between 0.06 and 0.11. MCC samples present a  
407 similar ability to compact while Lactose samples, having similar size distributions, present a  
408 quite different compaction behavior. Retalac sample, a co-processed dry binder, exhibit a  
409 lower packing density than Tablettose 70, generally attributed to the presence of  
410 Hypromellose and particles irregular shape. Food C and Food D have the higher packing  
411 density. But, no general conclusions can be drawn on the compaction behavior of food  
412 samples mostly attributed to the different nature of the particles.

413

414 From a global perspective as Ribiere et al., whose work focuses on a granular system  
415 composed of glass beads of 1 mm diameter [11], our experiments have established that all 9  
416 powders studied, when subjected to a tapping dynamics, compact and reach a stationary state

417 that depends on the tapping intensity (Figure 15). Moreover, all samples seem to reach a  
418 maximal compaction state during vibration. Powders such as Food D and Tablettose 70, after  
419 a maximal state of compaction, when increasing the vibration amplitude, the powder bed  
420 undergoes decompaction in a quite slow manner. This “slow decompaction process” allows to  
421 see the convection dynamic taking place at vicinity of the lateral walls and so, to follow  
422 progressively the decompaction process. For samples such as Food B and Food E,  
423 decompaction takes place in a quite abrupt way, generating a powder cloud making  
424 impossible to measure volume changes. *Decompaction dynamics seems to be time dependent,  
425 even more powder-cohesiveness dependent. More studies should be carry-out to fully fill this  
426 statement.*

427

428 Powders compaction behavior under free surface conditions can be schematized as presented  
429 in Fig.16, where three regions can be observed:

- 430 • Compaction (region I): the powder bed volume decreases sharply as the vibration  
431 amplitude increases. During this first step of compaction, a rearrangement of the powder  
432 particles occurs when the energy supplied to the bed powder overcomes interparticle  
433 forces.
- 434 • Maximal compaction (region II): no evolution of the powder bed volume is observed, and  
435 so a constant compactness state is observed. This region, could be interpreted as the  
436 maximal/densest compaction state of the powder bed, also known as the random close  
437 packing limit, where the particles are touching and packed in as tightly as possible. In this  
438 region, the powder has a solid-like behavior.
- 439 • Decompaction (region III): corresponds to the transition between solid-like and fluid-like  
440 behaviors. It begins with a dynamics of convection generating the auto-aspiration of air  
441 into the powder bed, and so inducing decompaction.

442

443 The HR was plotted as a function of the vibration amplitude for MCC, Lactose and Food  
444 powders as shown respectively in Fig.17, Fig. 18 and Fig. 19. For all figures, HR is only  
445 determined until the powder bed undergoes decompaction.

446 According to HR classification, MCC powders can exhibit fair, passable up to poor flow  
447 behavior depending on the vibration energy imposed to the sample (Fig. 17). The flowability  
448 of Avicel ph 105 is poorest than Avicel ph 102 ascribed to differences in moisture content or  
449 particle size distribution [20]. Our experiences validated this statement but showed an  
450 important dependency with the amplitude of vibration. Indeed, Avicel ph 105 has a better



451 flow behavior at low vibration amplitudes than Avicel ph 102 powders ( $A_0 < 1.6$  mm). When  
452 increasing the amplitude of vibration,  $A_0 > 1.6$  mm, Avicel ph 102 powders undergo  
453 decompaction while Avicel ph 105 samples continues to compact. According to Geldart's  
454 classification, cohesive materials have HR values greater than 1.4 [18]. Thus, Avicel ph 105  
455 cohesiveness could explain why the powder does not undergo decompaction even at high  
456 vibration amplitude/energy, as depicted in Fig. 15.

457

458 For lactose powders HR classification shows that Tablettose 70 samples exhibit a better  
459 ability to flow than Retalac (Fig. 18). The vibration amplitude range to obtain a steady HR  
460 value differs for Tablettose 70 ( $0.8 < A_0$  (mm)  $< 1.4$ ) and Retalac ( $1.5 < A_0$  (mm)  $< 3$ ).  
461 Moreover, as depicted from Fig. 18, Tablettose 70 undergoes decompaction at smaller  
462 amplitudes than Retalac, suggesting that Retalac particles possess stronger interparticle forces  
463 interaction.

464

465 Food Powders flow behavior from HR classification shows that before decompaction Food E  
466 and Food C samples exhibit a fair ability to flow while Food A, Food B and Food D samples  
467 exhibit a fair ability to flow (Fig.19).

468

469 The vibration amplitude, at a fixed frequency, is responsible for the amount of energy  
470 supplied to the powder bed (calculated from Eq. 9, shown in Fig. 20). *This vibration energy*  
471 *will allow the particles to jump and to reorganize, or not.*

472

473 When the powder bed is at its maximum compaction state, a maximal HR value is obtained.  
474 This maximal HR value, obtained under free surface conditions, is named in this work  
475 "**Ultimate Hausner ratio** ( $HR_U$ )". Corresponding to the maximum packing fraction of the  
476 powder,  $HR_U$ , is indeed a topological intrinsic characteristic of a powder, which can be used  
477 to understand, or even more to quantify, in a quite simple way, the role of powder  
478 interparticle forces in flowability.

479

480 In industrial applications, vibrations have been used together with fluidization in order to  
481 overcome cohesion problems [21, 22]. The quantification of the vibration energy needed to  
482 compact the powder bed, or to undergo decompaction, under free surface conditions, is of  
483 great interest in powders handling, filling and transport operations. Thus, the assessment of  
484  $HR_U$  could result in significant time and resource saving in industrial technology.

485

486 3.2.4. Flowability results comparison

487

488 A compilation of HR values obtained with the 3 devices used in this work, Densitap®,  
489 Granupack® and our Vibration device, is gathered in Table 7. For simplicity, we chose to  
490 compare between HR values obtained by free-falls performances at 3 mm, for Densitap® and  
491 Granupack® devices, with the values obtained with our Vibratory device, using as reference  
492 the Ultimate Hausner ratio (HR<sub>U</sub>).

493

494 **Table 7**

495 Hausner ratio according to Densitap®, GranuPack® ( $A = 3$  mm) and Vibratory device

Powders	HR (-) DensiTap®	HR <sub>∞</sub> (-) GranuPack®	HR <sub>U</sub> (-) Vibratory device
Tabletose70	1.14 ± 0.01	1.25 ± 0.03	1.21 ± 0.02
Food D	1.15 ± 0.02	1.27 ± 0.01	1.27 ± 0.01
Food E	1.21 ± 0.01	1.14 ± 0.02	1.19 ± 0.01
Food C	1.22 ± 0.02	1.20 ± 0.01	1.18 ± 0.01
Food A	1.25 ± 0.01	1.31 ± 0.02	1.27 ± 0.01
Retalac	1.26 ± 0.02	1.24 ± 0.01	1.31 ± 0.02
Food B	1.28 ± 0.03	1.28 ± 0.02	1.29 ± 0.02
Avicel ph 102	1.31 ± 0.04	1.27 ± 0.01	1.35 ± 0.01
Avicel ph 105	1.44 ± 0.04	1.41 ± 0.01	1.44 ± 0.01

496

497

498 To facilitate the analysis two figures are presented. In Fig.21 are compare the HR values  
499 obtained with DensiTap® and GranuPack®. As depicted from the figure, from nine powder  
500 samples studied only five possess statistically similar values. Indeed, samples such as  
501 Tabletose 70, Food A, Food D and Food E presented a quite different flowability  
502 classification.

503 No correlation between particle's size, shape and true density were found that allowed to  
504 explain all HR differences obtained. For example, for the heaviest particles, Food B

505 (2.28 g/ml), HR values obtained with both devices are statistically similar, while for the  
506 lightest particles, Food E (1.23 g/ml), this correlation is no longer valid, suggesting a density  
507 dependency. Nevertheless, for the other seven powder samples with true density values  
508 between 1.42 and 1.57 g/ml non correlation can be done. Similarly for size- and shape-based  
509 analysis, [no correlation has been found](#).

510

511 Upon impact, during tapping, a serial of physical phenomena takes place allowing wave  
512 propagation into the powder bed. Thus, despite similar free-falls height performances it is  
513 possible, even logical, to assume that the energy provided to the powder bed is device  
514 dependent, mostly attributed to materials properties. Under this perspective, from Fig 21. it  
515 seems that GranuPack® device supplies more energy to the system, but even here, no general  
516 conclusions can be drawn (Food E). Without any measurements allowing to quantify the  
517 amount of energy supplied to the powder samples, one point that could perhaps explain this  
518 HR differences is the presence of a “diabolo” in GranuPack® device. The “diabolo”,  
519 deposited on the surface of the powder bed, is used in order to measure, by means of a sensor,  
520 the height of the powder bed after each tap. Diabolo’s presence could change, mostly for low  
521 density particles, the stress patterns into the powder column and so could impact powders  
522 reorganization and finally the HR values obtained. Furthermore, it doesn’t allow free surface  
523 reorganization. Thus, to avoid any misconception caused by diabolo’s impact no other  
524 comparison using GranuPack® HR values is presented.

525

526 In Fig. 22, HR values obtained with our Vibratory device are compared with those obtained  
527 with DensiTap®. As depicted in the figure, HR values obtained with the Vibratory device are  
528 higher than those obtained with DensiTap®. These results validate our statement relating  $HR_U$   
529 to a maximal state of compaction energy-dependent. Thus, the energy supplied to the powder  
530 bed during harmonically driven motion, as in particle dampers, can reach higher values than  
531 those obtained in fixed free-fall height devices. Other experiments allowing to measure the  
532 energy supplied to the system in this kind of devices should be carried out for validation.

533

534 In the case of Food E and Food C samples,  $HR_{DensiTap®}$  values are higher than  $HR_U$  values.  
535 These two exceptions can be attributed to powders good ability to flow (Fig 20). Indeed, the  
536 assessment of powders flowability by compaction techniques is not suitable for good  
537 flowability powders because of their difficulties to compact. During vigorous vibration, at

538 high energy, just before an abrupt decompaction, the aeration of the powder bed can take  
539 place, inhibiting the system to reach a maximal compaction state.

540

541

542

543

### 544 *3.3. General discussion: a critical view*

545

546 Non general-conclusions can be drawn relating flowability to the size distribution or the  
547 morphology (shape) of the powders. This is mostly attributed to the complexity of granular  
548 materials used in our work. The heterogeneity of our powder samples in terms of nature,  
549 shape, size distribution and physical properties (crystallinity, density, roughness, porosity...)  
550 makes quite difficult the analysis enabling to draw any general statement.

551

552 As Ribiere et al., 2007, we have established that non-ideal powders (non-spherical, large size  
553 distribution, cohesive ...) subjected to tapping compact (packing fraction increases) and reach  
554 a stationary state that depends on the tapping intensity.

555

556 The study of metal powders or glass beads flowability using bulk densities ratio has been  
557 successfully achieved [6, 23, 24]. When referring to more complex samples, such as organic  
558 powders, numerous limitations has been highlighted when using HR to evaluated flow  
559 behavior such as low reproducibility, user-dependency, scattered results, amount others [4,  
560 16]. Determining powders flow behavior using criteria such as HR, implies mastering many  
561 parameters, among them the powder mechanical history, that is to say the different  
562 mechanical stresses supplied to the powder. In this study, we have shown that HR is strongly  
563 related to the energy injected to the powder bed. The system approaches its optimally packed  
564 state for a given vibration intensity but not seems to be user-dependent or to present low  
565 reproducibility (Fig. 14 and 20). Classic vibration compaction devices such as Densitap  
566 ( $A = 3$  mm and  $f_t = 4.16$  Hz) and Granupack ( $A = 1$  or  $3$  mm) supplied a quite fixed vibration  
567 energy to the powder samples under study. If this energy is not enough to allow the particles  
568 to reorganize, the HR value obtained will not be an accurate representation of the powder  
569 ability to flow.

570

571 Over 50 years later, Hausner reflection is still valid. During vibration the powder particles are  
572 forced to jump, and those to lose contact with each other for a moment. During this time there  
573 is no friction, between the particles. During this frictionless moment, the particles are able to  
574 rearrange and thus to compact [6]. **What has not been taken into account, is the amount of**  
575 **energy needed to actually allow the particles to jump and rearrange.** What we define as the  
576 **HR<sub>U</sub>**, is indeed, the bulk density ratio allowing the maximal powder  
577 rearrangement/compaction; refers to the elastic deformation of the particles and therefore **is**  
578 **an intrinsic characteristic of the powder** i.e. **independent of the tester used.** In fact, the  
579 maximal reorganization/rearrangement state of a powder, without any particles deformation  
580 can be achieved, or not when using a vibratory device under free surface conditions. The  
581 maximal reorganization state of a powder in a vessel is intrinsic to the powder. If the device  
582 used to estimate flowability does not allow to supply to the powder bed the energy needed to  
583 compact until its maximal compaction state, the system will not achieve the maximal  
584 reorganization but it will achieve a given compaction state. The higher the injected energy is,  
585 the more important the structural modifications are, in agreement with compacity fluctuations.  
586 Indeed, this input energy allows to move the system from one equilibrium state to another  
587 one. Thus, the powder compaction state depends on the amount of energy supplied to the  
588 system while the amount of energy will depend on the device physical and operating  
589 characteristics (amplitude and frequency of the vibration, system materials, ...) but the  
590 maximal compaction state is not device dependent.

591  
592 The HR<sub>U</sub>, obtained at the maximum packing fraction of the powder bed, allows to relate HR  
593 to an intrinsic parameter of the system. Thus, HR<sub>U</sub> will provide to some extent a better  
594 assessment of a powder flow behavior during granular compaction.

595  
596 The data presented in this article are mainly given in order to stimulate additional work on the  
597 subject which will result in a better understanding of the flowability of powders.

#### 598 599 **4. Conclusions**

600  
601  
602 Over the last several years, Hausner Ratio, and the closely related Carr Index, have become  
603 quite popular to evaluate powders flowability, despite the several limitations involved  
604 because of the simplicity and rapidity of the [measure](#).

605 In order to study the consistency of HR with flow, three vibratory compaction devices were  
606 used. Different types of powders were examined, used in pharmaceutical and food  
607 industries, to cover a large range of applications.

608

609 As expected, the assessment of HR with DensiTap®, GranuPack® and our vibratory device,  
610 revealed at which point HR values can be modified as function of the dynamic parameters  
611 used. The amplitude and the frequency of the vibration, and thus the energy supplied to the  
612 system seems to be the major responsible of these fluctuations. A new approach is proposed  
613 to determine flow behavior by using HR based in the study of the energy supplied to the  
614 powder bed.

615 A number of observations can be reported regarding to the obtained results:

- 616 (a) The stationary state does not depend on the initial conditions of the powder bed;
- 617 (b) Non-ideal granular packings, as the ones used in this work, submitted to mechanical taps  
618 or sinusoidal cycles, can reach a stationary state that depends on the tapping intensity at a  
619 given frequency;
- 620 (c) HR obtained at the stationary state showed to be strongly dependent on the energy applied  
621 to the system. Indeed the input energy is related to the device used and so, on the  
622 experimental conditions such as the vibration amplitude;
- 623 (d) The energy supplied to the system will allow, or not, the reorganization of the grains  
624 (packing fraction increases);
- 625 (e) To obtain a HR value in consistency with the powder flow behavior, it seems necessary to  
626 determine bulk densities at the stationary state but also under a maximal compaction state.  
627 This value is named “**ultimate HR, (HR<sub>U</sub>)**”;
- 628 (f) Classical devices, fixed free-fall height, do not always allow to determine **HR<sub>U</sub>**, because of  
629 energetic limitations;
- 630 (g) The **HR<sub>U</sub>** value is an intrinsic property of the material and could be related the powder  
631 cohesiveness.

632

633 Finally, there is no doubt that HR, as a factor so simple to measure, has a great potential to  
634 determine flow behavior when taking into account the energy supplied to the system. The  
635 understanding of the effects of physical and mechanical properties on powders behavior can  
636 decrease the need to perform powder flowability analysis, resulting in significant time and  
637 resource saving.

638 The relation between **Ultimate HR** and interparticle forces interaction is the subject of a  
639 separate paper currently in preparation.

640

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645

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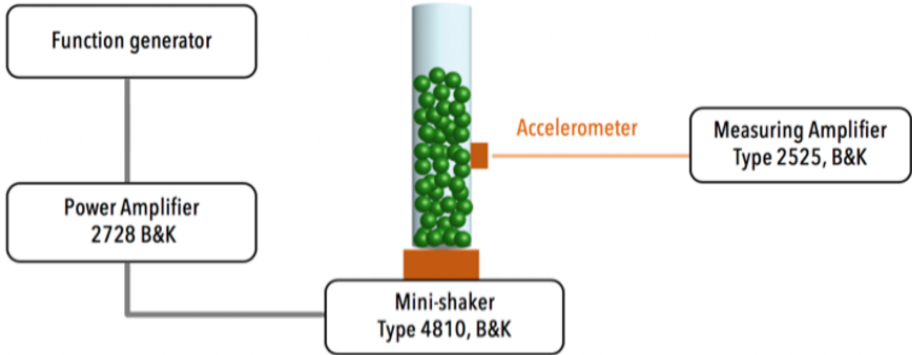
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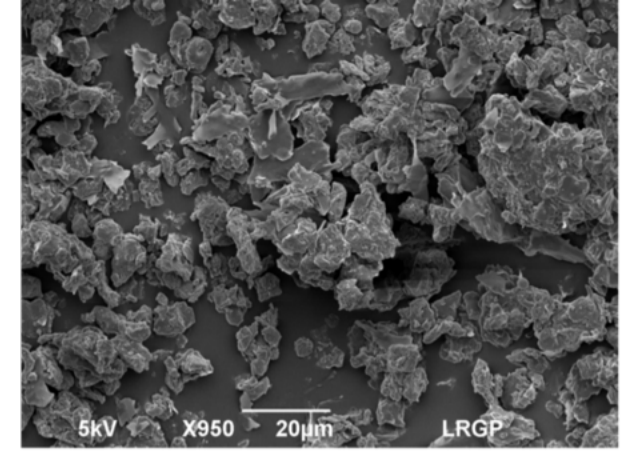
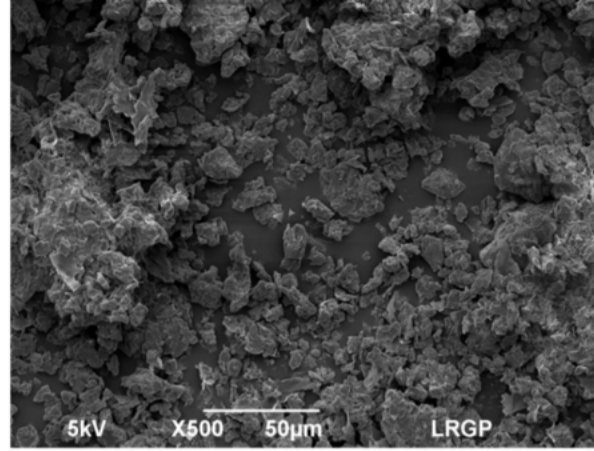
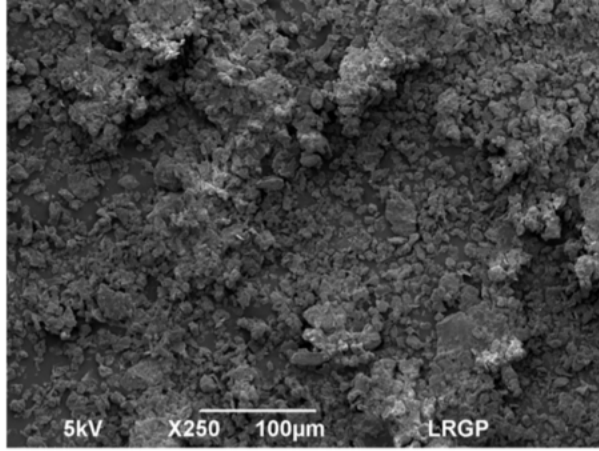
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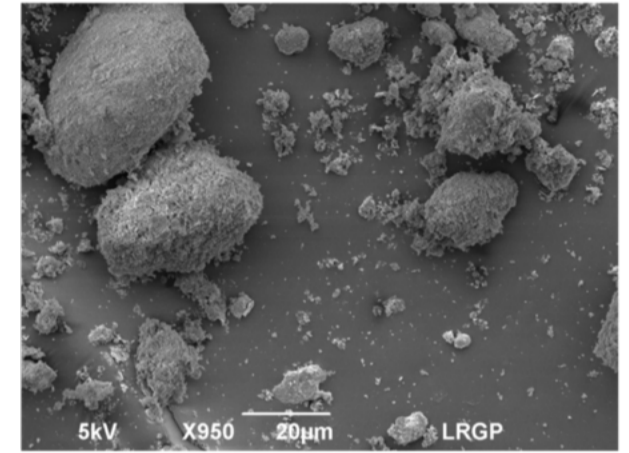
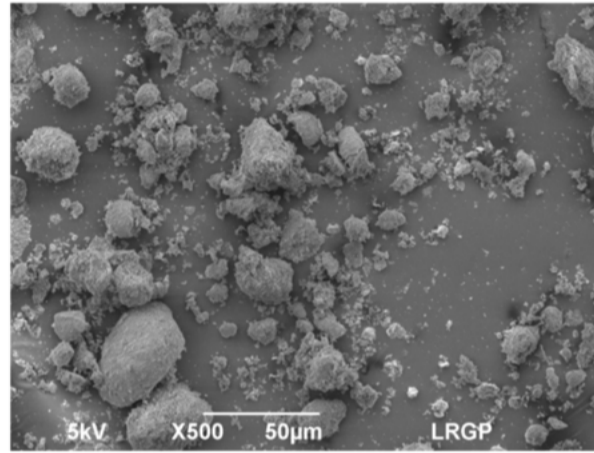
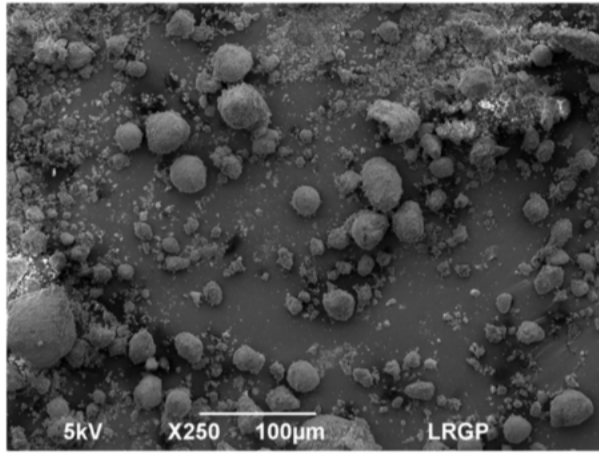




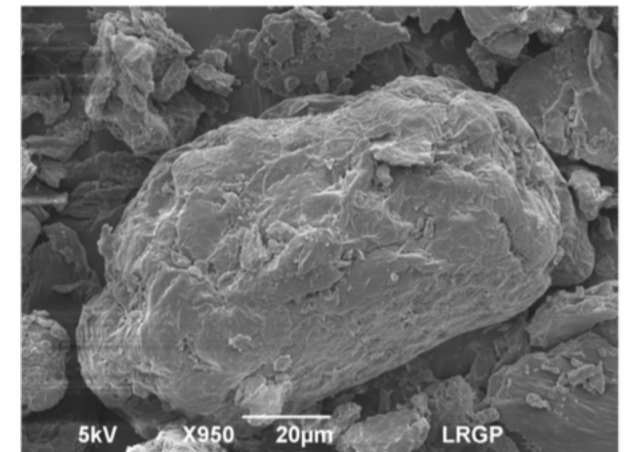
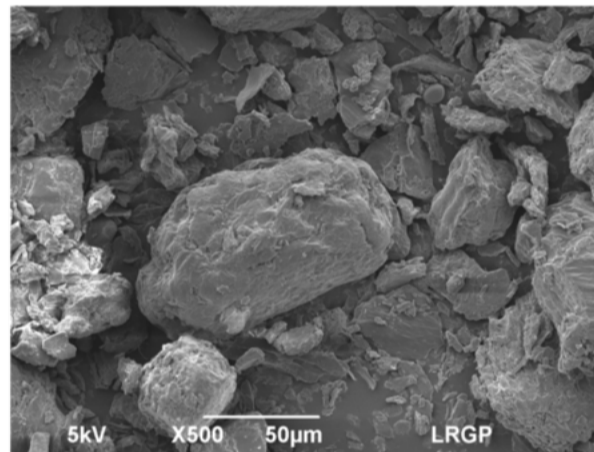
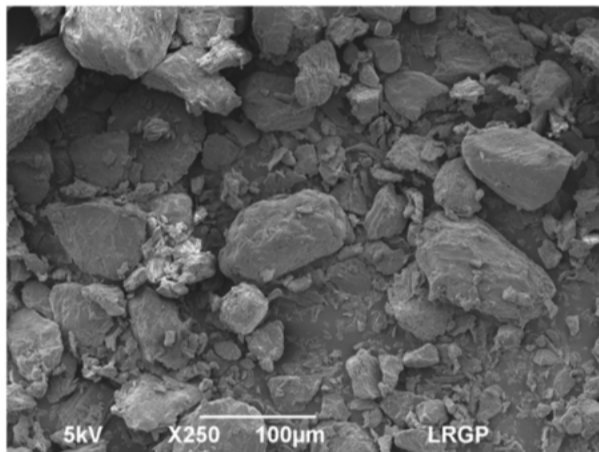
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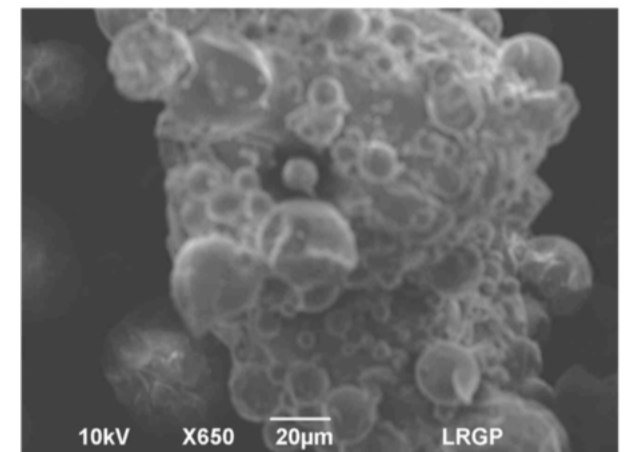
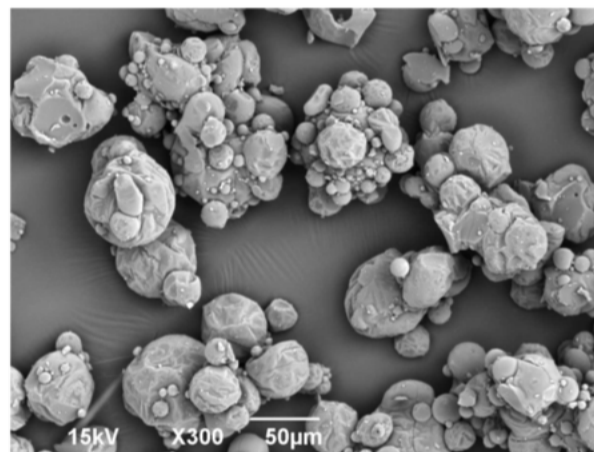
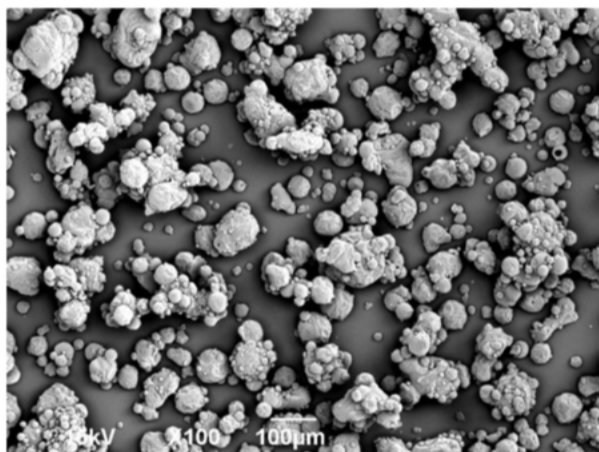
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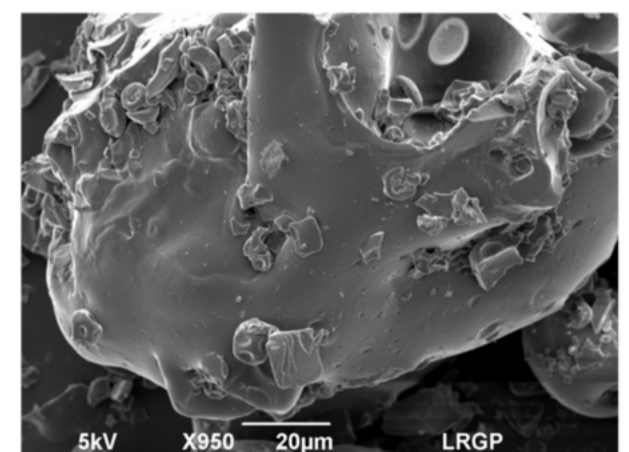
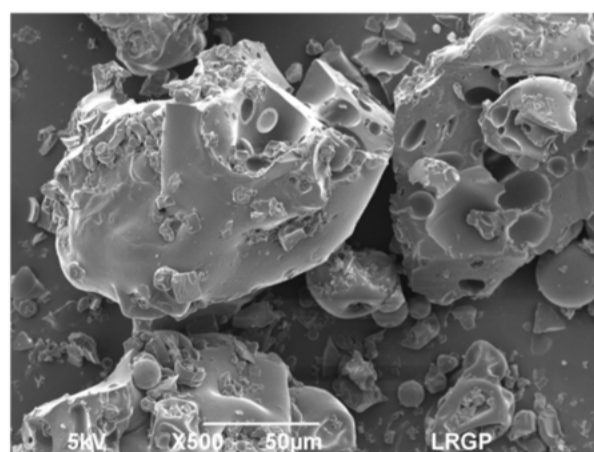
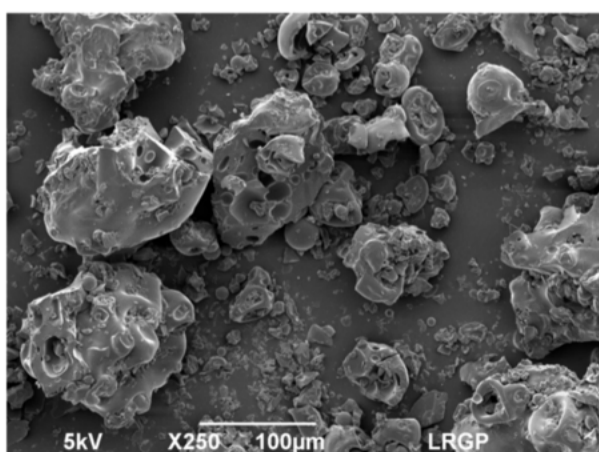
Food C



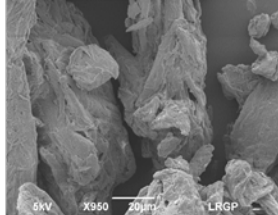
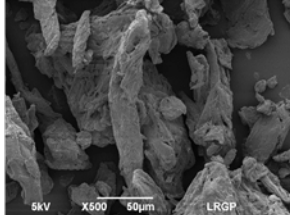
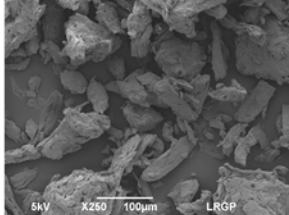
Food D



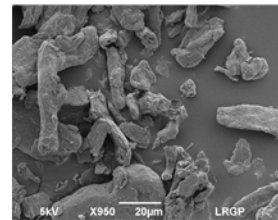
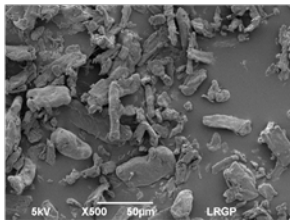
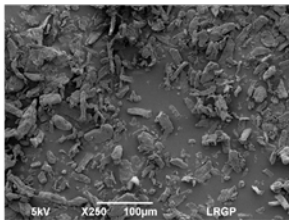
Food E



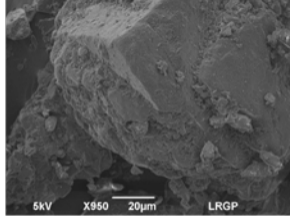
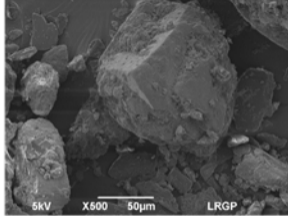
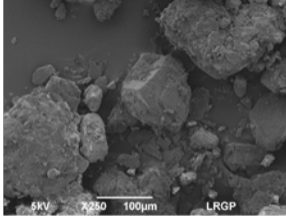
Avicel ph 102



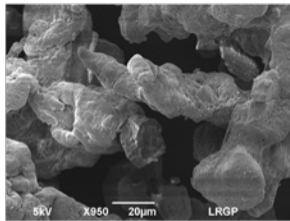
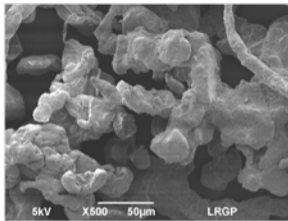
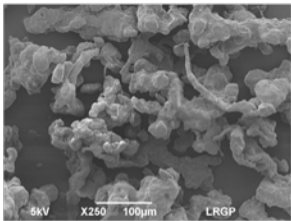
Avicel ph 105

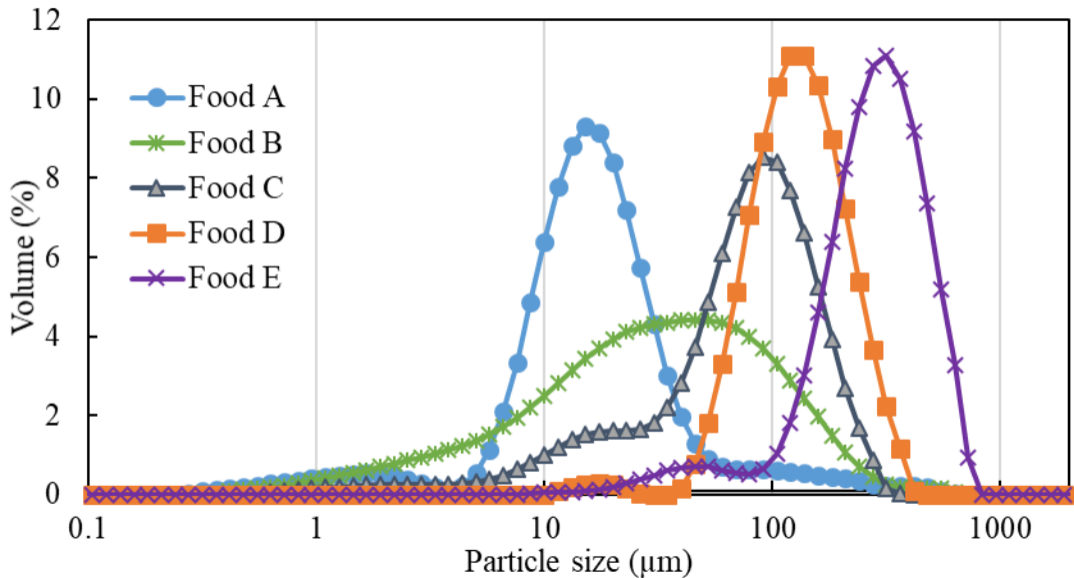


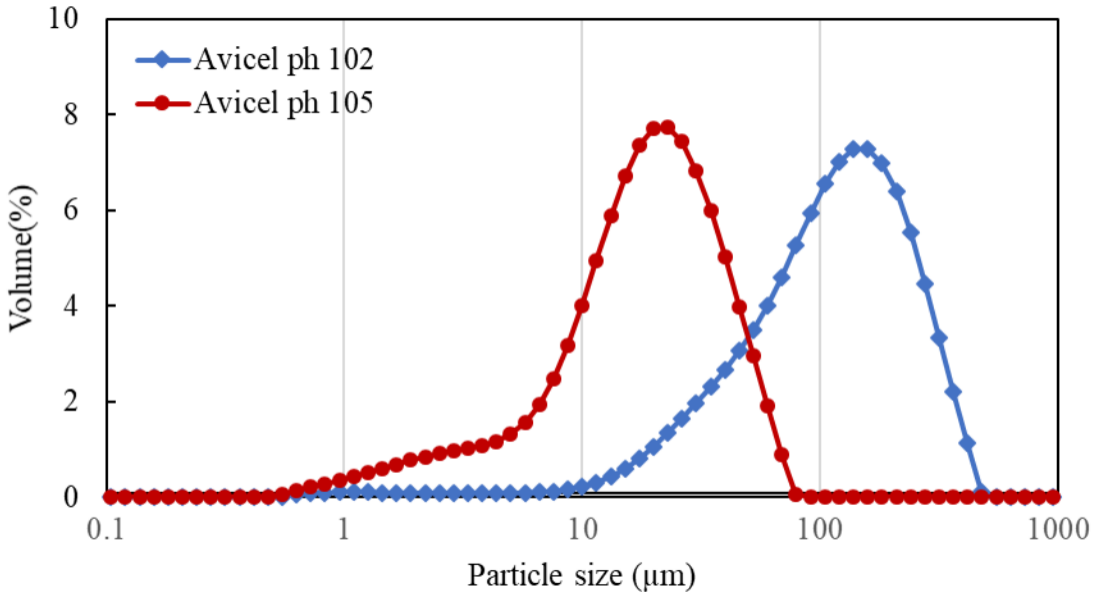
Tabletose 70

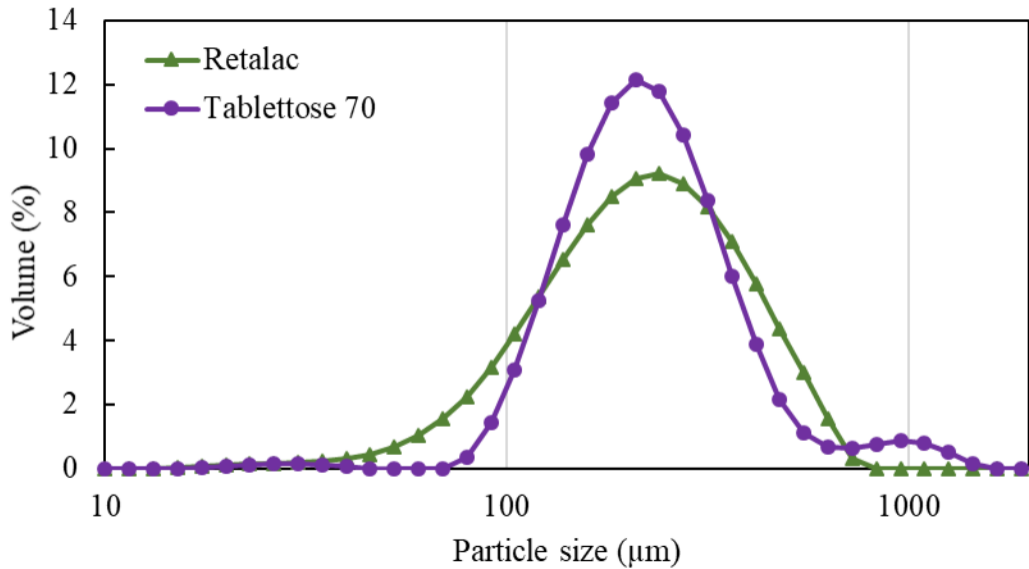


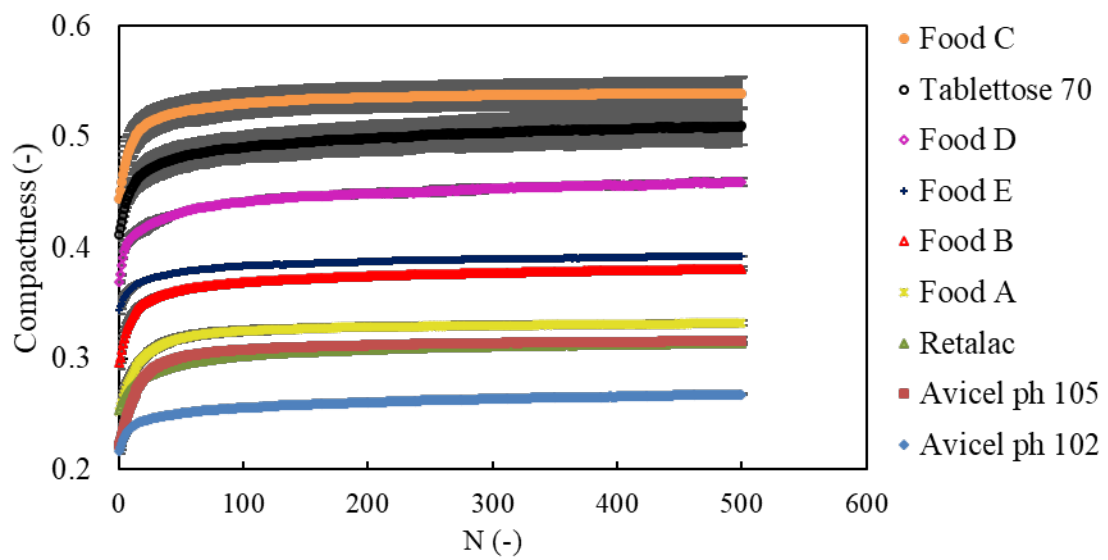
Retalac



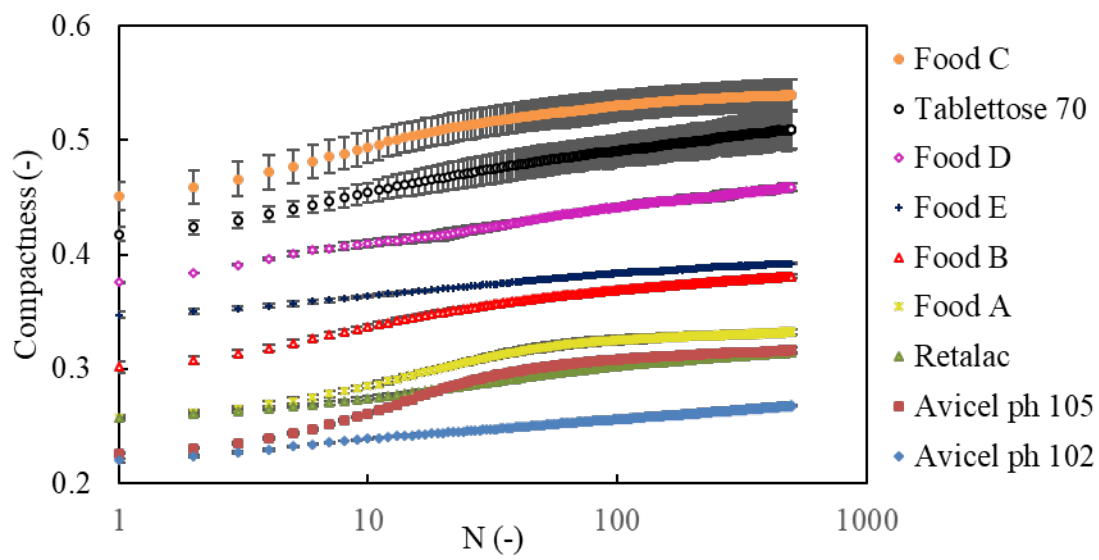


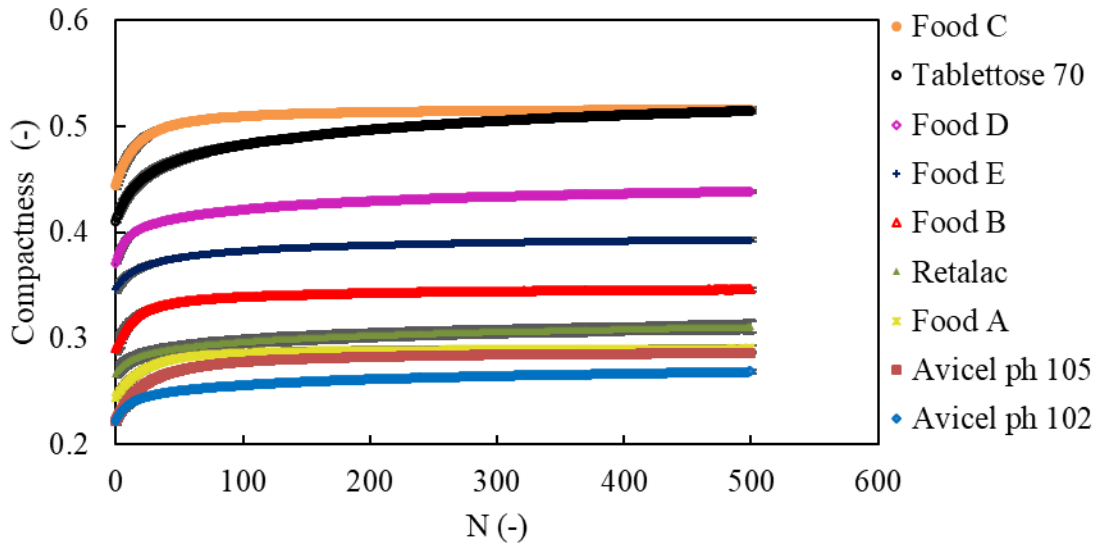


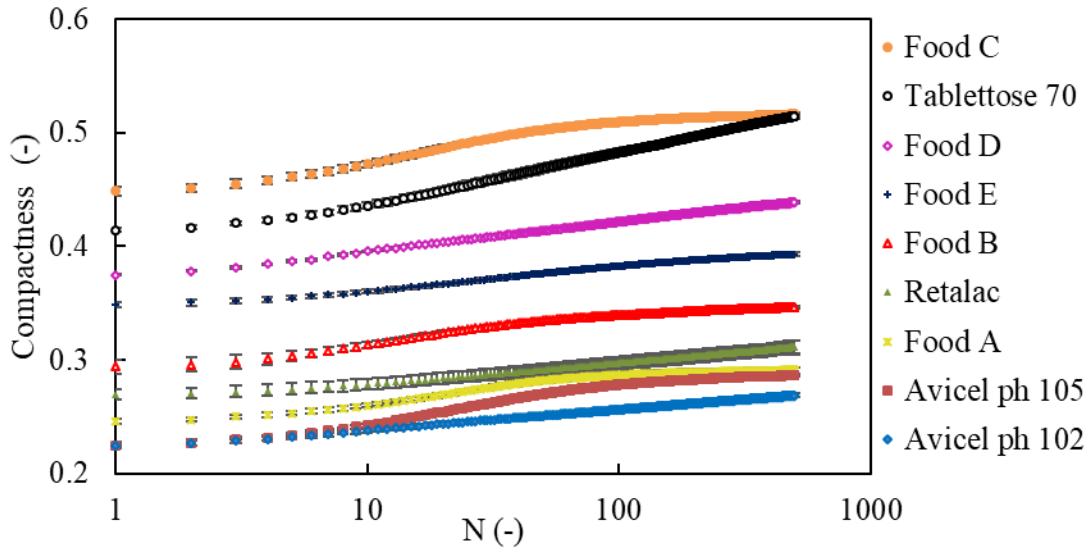


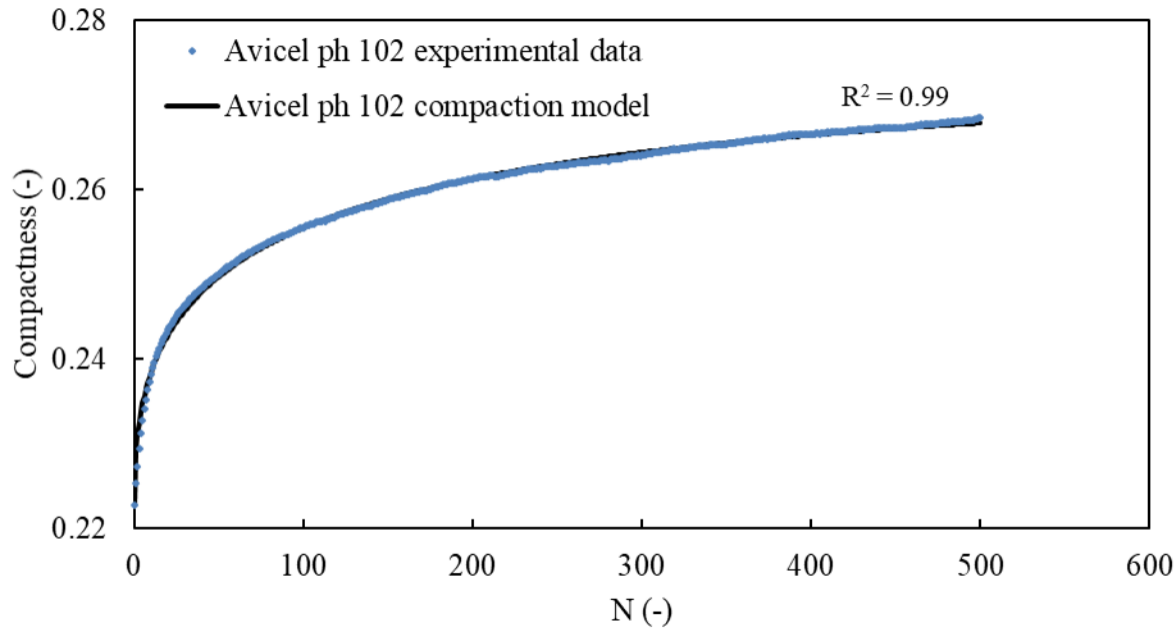


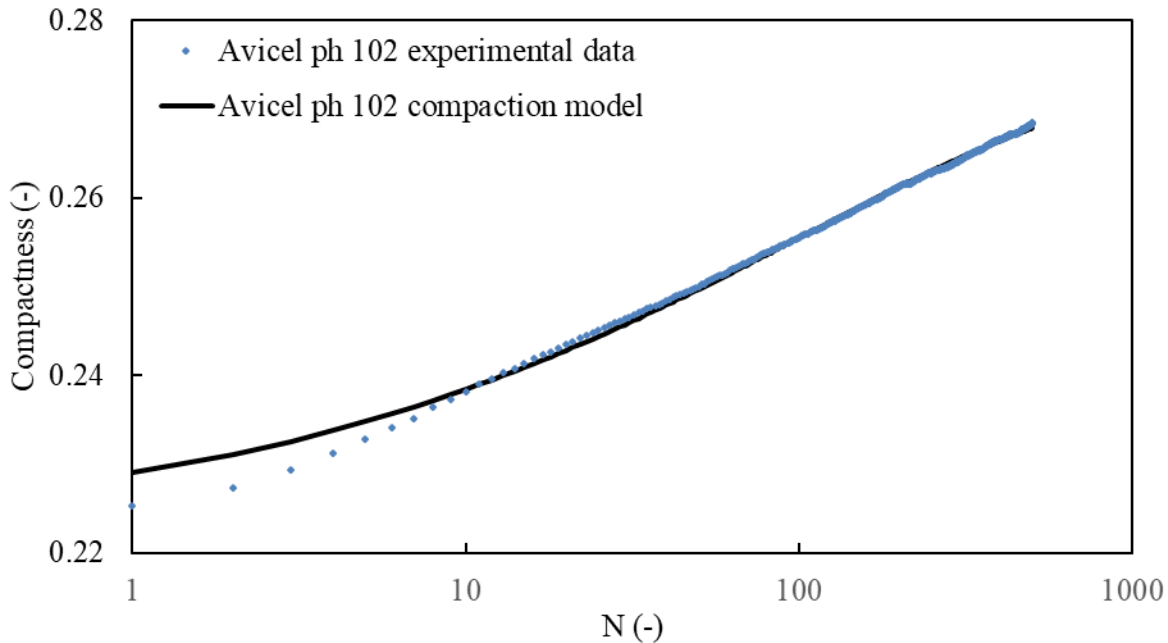




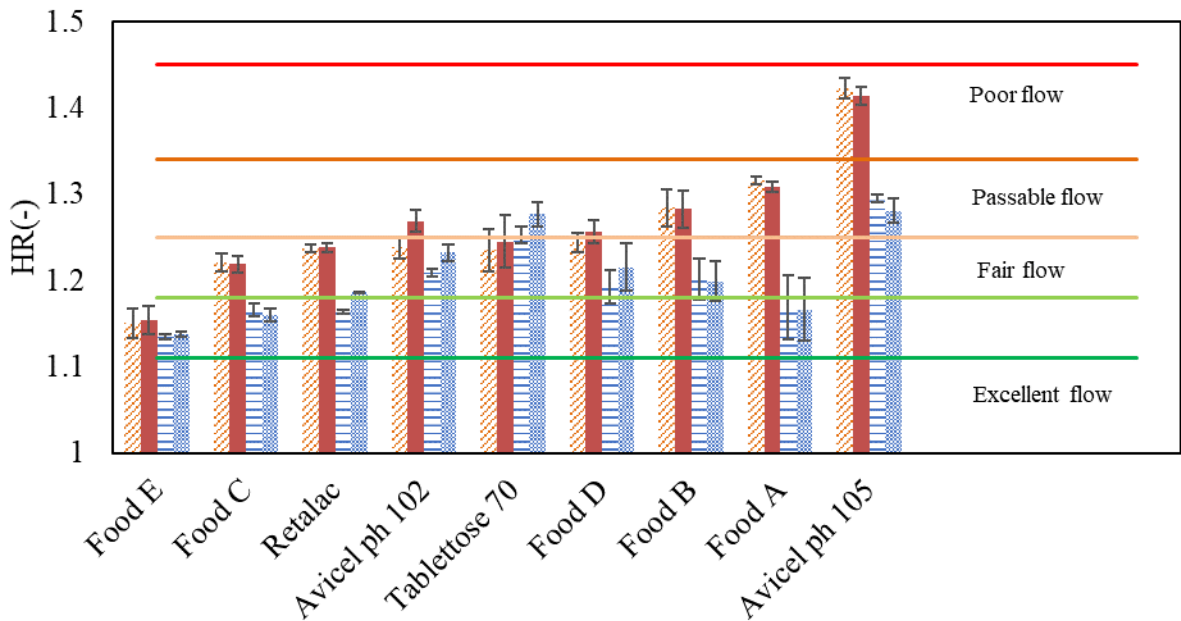


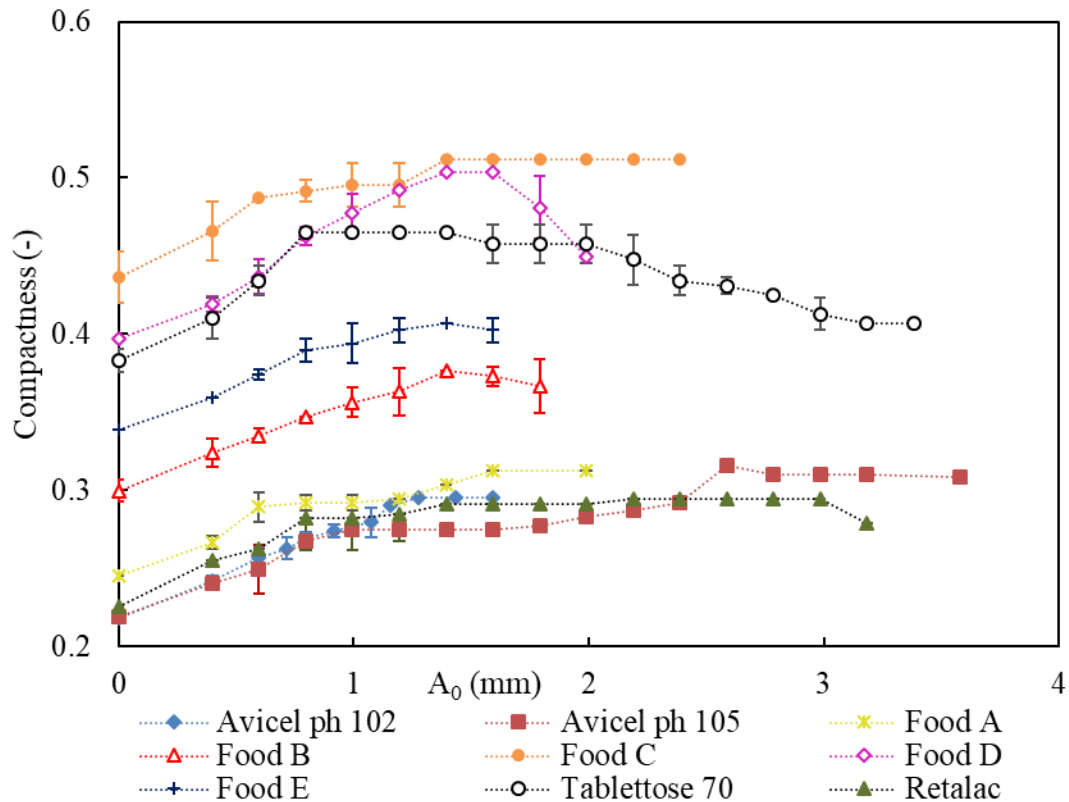


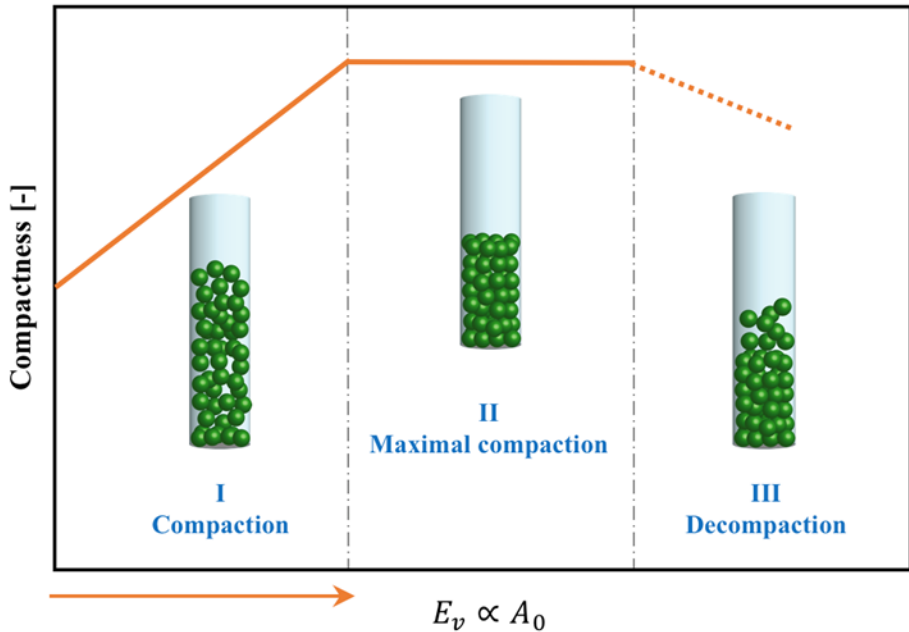




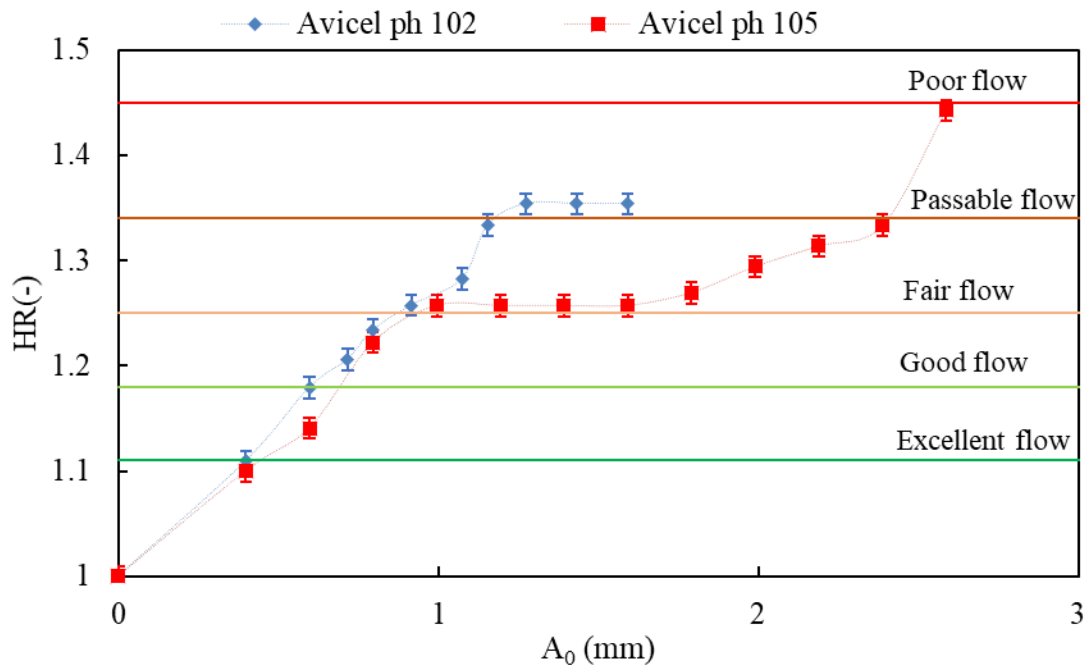
▨ A = 3 mm HR (500)   ■ A = 3 mm HR( $\infty$ )   ▨ A = 1 mm HR(500)   ▨ A = 1 mm HR( $\infty$ )



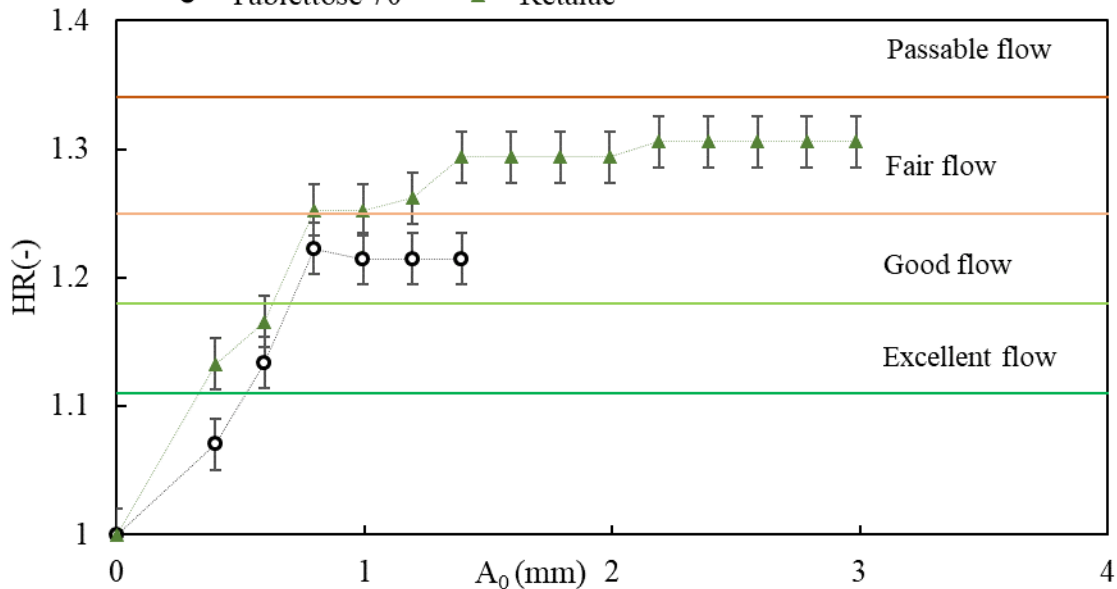


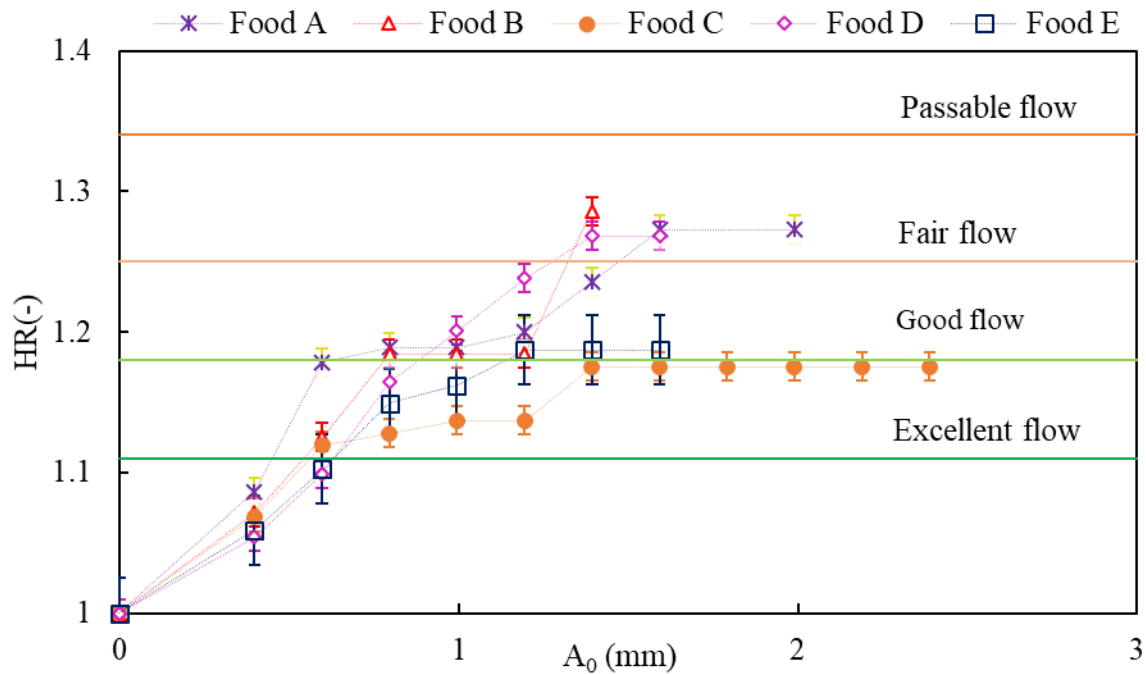


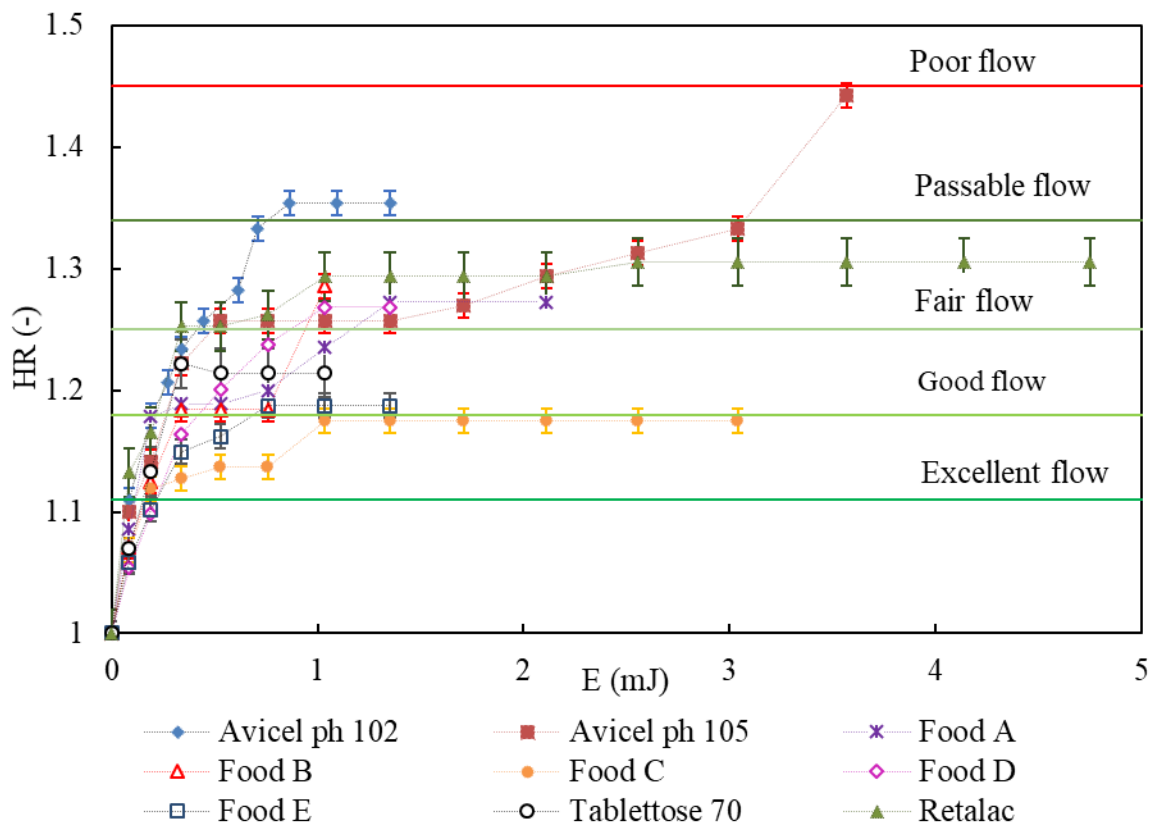


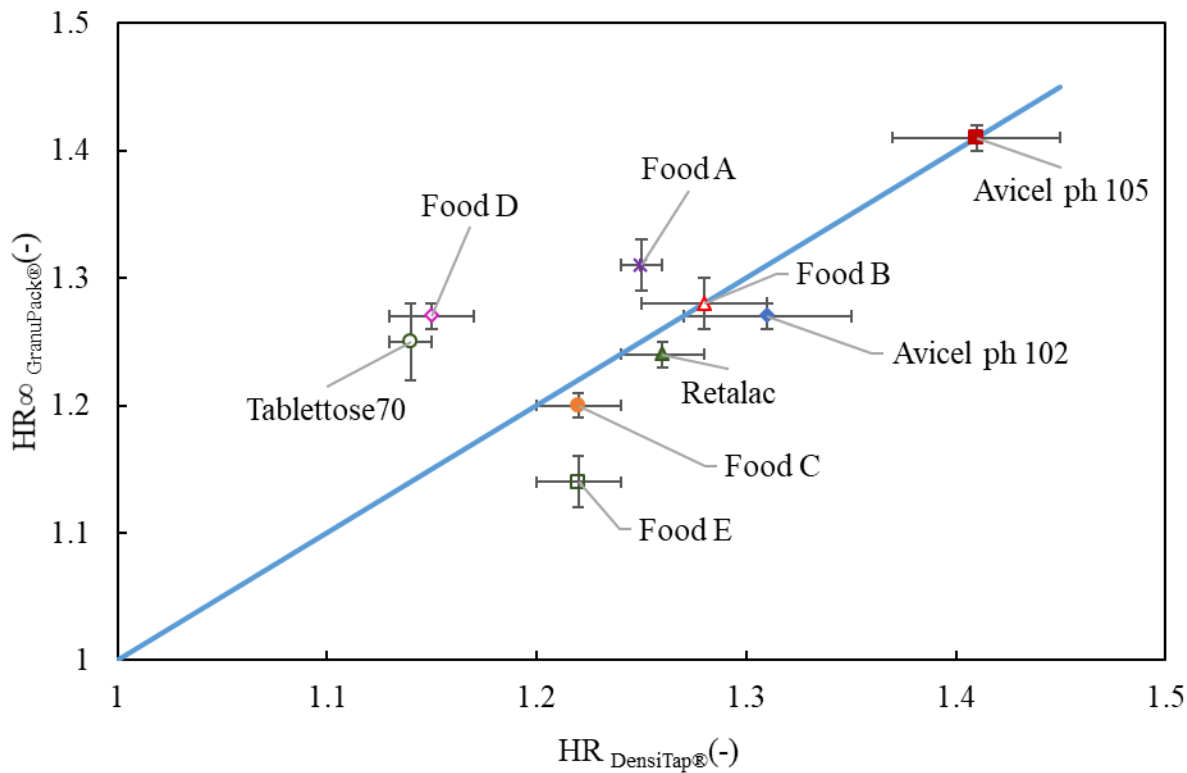


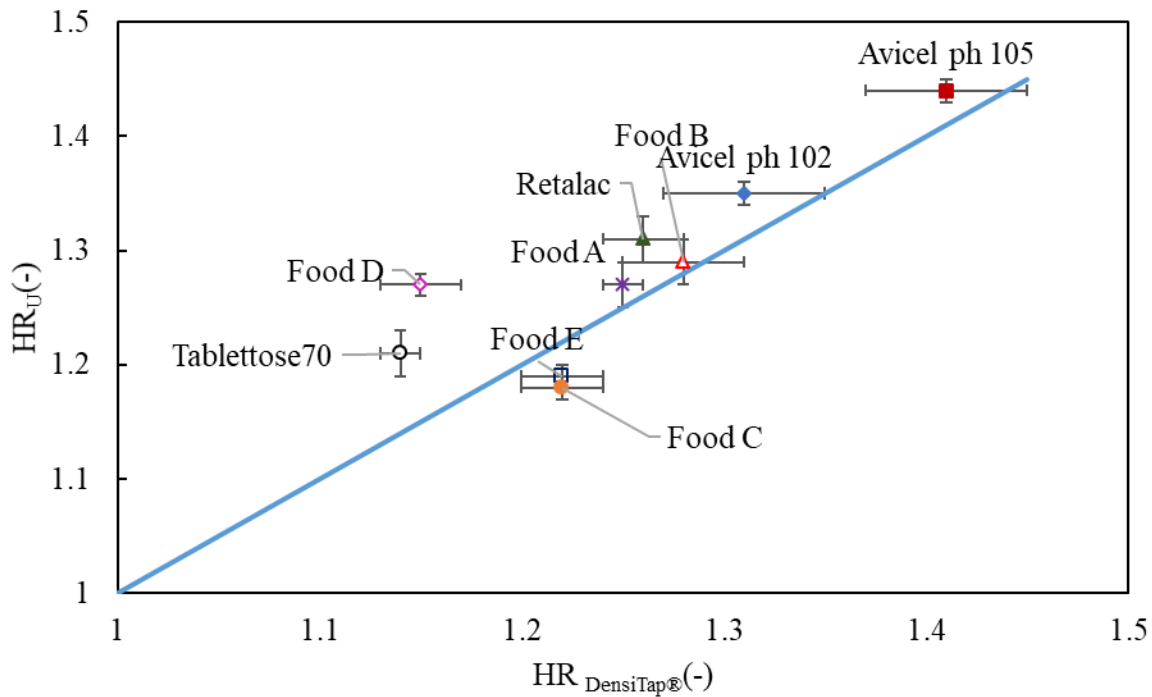
● Tablettose 70    ▲ Retalac



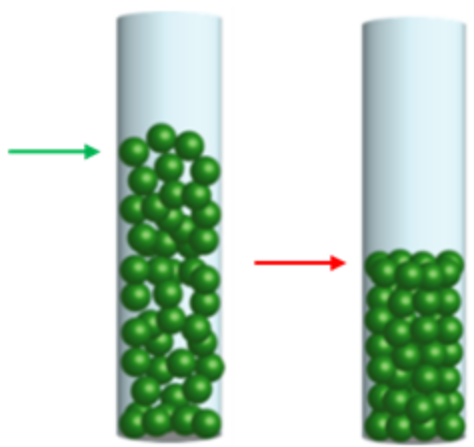








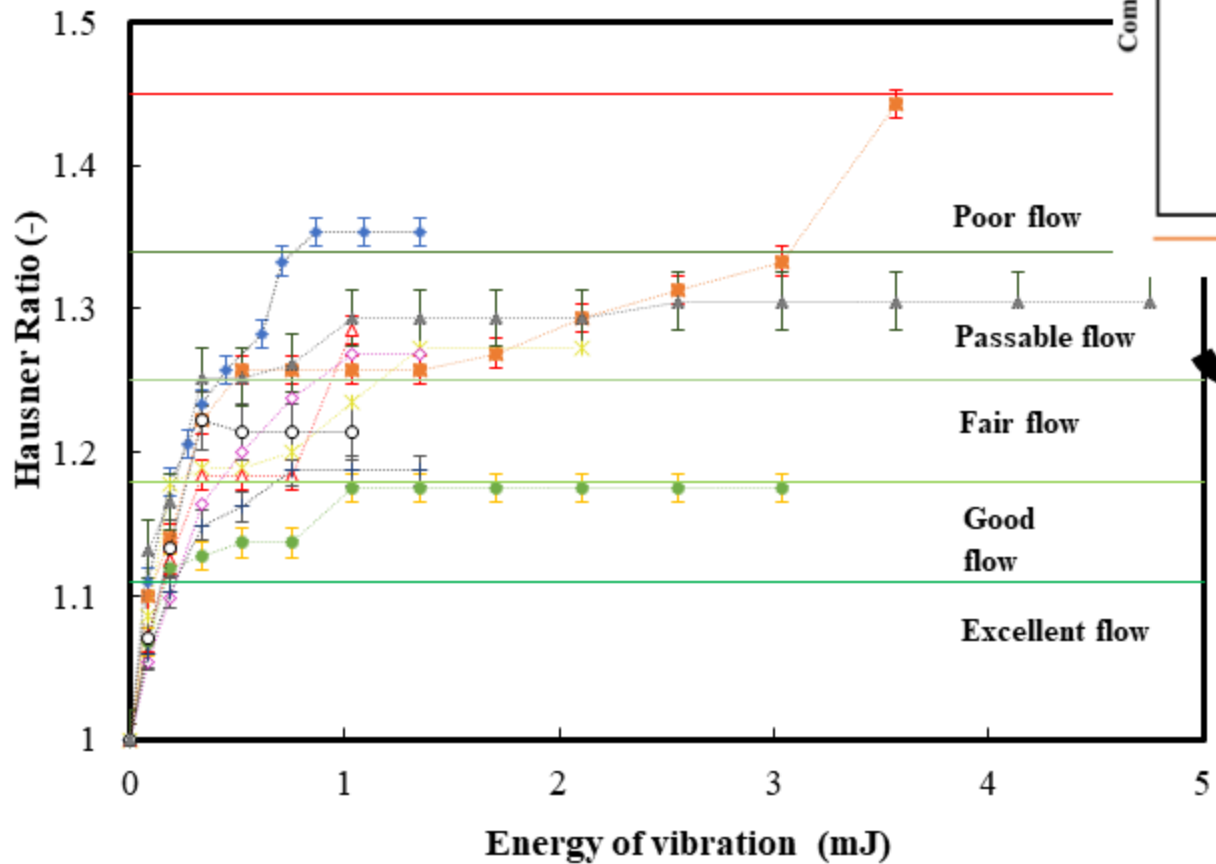
$$HR = \frac{\text{initial volume}}{\text{final volume}}$$



Shaker

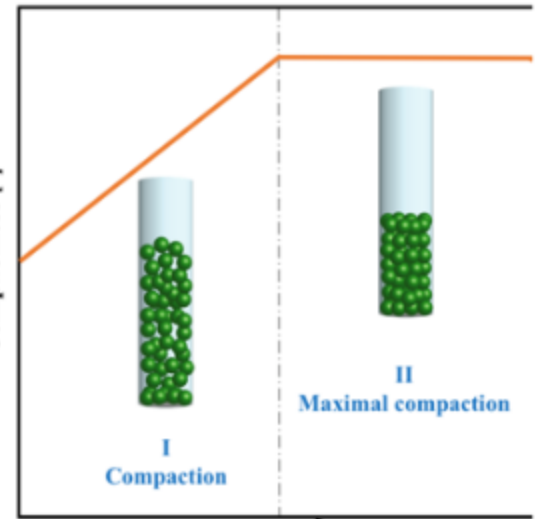
Harmonic vibration

$$E_v = \frac{1}{2} m (2\pi f)^2 A_0^2$$



- ◆ Avicel ph 102
- Avicel ph 105
- ✱ Food A
- ▲ Food B
- Food C
- ◇ Food D
- + Food E
- Tablettose 70
- ▲ Retalac

Compactness [-]



$$E_v \propto A_0$$