

## 수분활성도를 달리한 모형식품분말의 압축성 및 응력이완

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### Compressibility and Relaxation Patterns of Model Food Powder at Different Water Activity Levels

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#### Abstract

Bulk flow properties of five kinds of model food powder composed of potato starchy powder (potato starch) and proteinaceous powder (wheat protein) were determined at different levels of water activity. Fitted equations relating the bulk properties to water activity were also developed. As either the water activity or the protein content of model food powder increased, compressibility, compaction ratio, and irrecoverable work increased; however, asymptotic modulus and relaxation (%) of the powder decreased. In all fitted equations the coefficients of determination were high enough (above 0.98) to guarantee the reliability of the equations.

Key words: compressibility, relaxation, model powder, water activity

#### Introduction

The bulk flow properties of food powder which play an important role in processing, handling, and storage may often be influenced by an environment surrounding them. Especially, relative humidity (or equilibrium water activity) strongly influences the bulk properties, causing flow difficulties or caking of powder (Peleg, 1977, 1983).

Extensive research works on the bulk flow properties are available in the literature (Mannheim and Passy, 1981). A few reports have been focused on the important bulk properties such as compressive deformation patterns (Hollenbach, 1982, 1983) and stress relaxation patterns (Hollenbach and Peleg, 1982). Such works have dealt with coffee (Moreyra and Peleg, 1980), coffee creamer (Peleg *et al.*, 1982), baby formula (Moreyra and Peleg, 1981), sugar (Hollenbach and Peleg, 1983),

onion powder (Moreyra and Peleg, 1980), bran powder (Moreyra and Peleg, 1982), soy protein (Hollenbach and Peleg, 1983), corn starch (Hollenbach and Peleg, 1983), anticaking agents (Hollenbach *et al.*, 1982), and glass beads (Peleg *et al.*, 1982, Scoville and Peleg, 1981).

Food powder often exists in the form of mixture composed of basic constituents and therefore the bulk properties may be affected by the chemical nature of each constituent powder. In general, starchy powder (e.g., potato starch and corn starch) is considered to be relatively free flowing, while proteinaceous powder (e.g., soy protein and wheat protein) relatively more cohesive. The bulk flow properties of each constituent powder may be utilized to predict or estimate the properties of mixed food powder. However, no attempts have been made to determine the bulk flow behavior, especially compressive deformation patterns and stress relaxation patterns of model food powder mixed with starchy powder and proteinaceous powder in an appropriate ratio. In addition, few of the re-

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ported properties have been related with the equilibrium water activity, which is the most influential to the flow property changes of food powder.

Therefore, in this research the compressive deformation patterns and the stress relaxation patterns of model food powder composed of starchy powder (potato starch) and proteinaceous powder (wheat protein) were determined and compared at different water activity levels, and the fitted regression equations relating those properties to water activity were developed.

## Materials and Methods

### Materials

Commercial potato starch and wheat protein were purchased at local stores. Five kinds of model food powders were prepared according to the mixing ratio as shown in Table 1.

### Determination of water activity

Water activity was measured with an electrohygrometer (Novasina Co., Model EE3A-3) after equilibration at 20°C.

### Determination of compressibility, compaction ratio, and irrecoverable work

Compression tests were carried out based on the procedures described in Peleg *et al.*, (1982), Scoville and Peleg (1981), and Moreyra and Peleg (1980). Fig. 1 shows the test cells used for the compression test. Powder was poured into the stainless steel test cell (45 mm in diameter and 30 mm in depth) and a cover plate (40 mm in diameter and 3 mm in thickness) was placed on the top of the powder. The test cell was then mounted

on the base plate of Instron Universal Testing Machine (Model 1000) and the powder specimen was compressed with a 50 kg compression load cell at a crosshead speed of 2.0 mm/min. The force-time curves were recorded at a chart speed of 40 mm/min.

Since the initial weight of the powder and the dimensions of the cell were known, the force-time curves could be transformed into bulk density-compressive stress (1-10 kg/cm<sup>2</sup>) relationships. The relationships could be described by the following empirical equation (Peleg, 1983, Peleg *et al.*, 1982).

$$\rho = a + b \log \delta_N \quad (1)$$

where:  $\rho$  = bulk density (g/cm<sup>3</sup>)

$\delta_N$  = compressive stress (kg/cm<sup>2</sup>)

a, b = constants

The constant "b" in Eq. (1) defined as the compressibility was calculated from the slope of semi-logarithmic plot. The constant "a" becomes a bulk density after compression at 1 kg/cm<sup>2</sup>. Compaction ratio can be expressed as the ratio of bulk density at the compressive stress of 1 kg/cm<sup>2</sup> to bulk density before compression (Scoville and Peleg, 1981, Moreyra and Peleg, 1980).

$$C_R = \frac{\rho_c}{\rho_0} \quad (2)$$

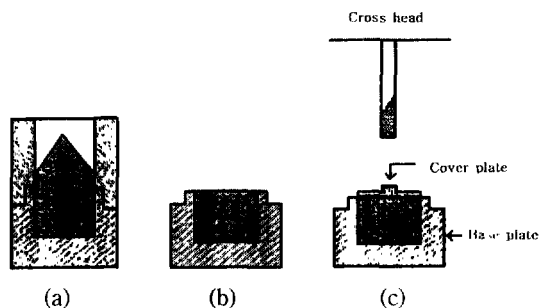
where:  $C_R$  = compaction ratio

$\rho_0$  = bulk density before compression (g/cm<sup>3</sup>)

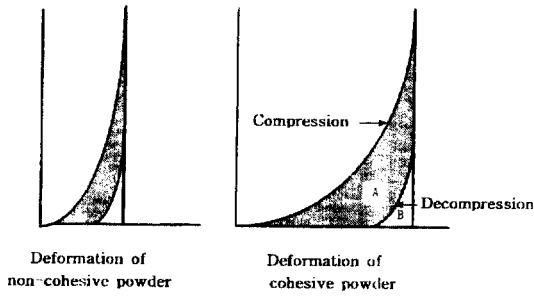
$\rho_c$  = bulk density after compression (g/cm<sup>3</sup>) at 1 kg/cm<sup>2</sup>

**Table 1. Formulations of five kinds of model food powder**

Model food powder	Mix ratio (based on w/w%)	
	Potato starch (Starchy powder)	Wheat protein (Proteinaceous powder)
S <sub>10</sub> P <sub>0</sub>	100	0
S <sub>7</sub> P <sub>3</sub>	70	30
S <sub>5</sub> P <sub>5</sub>	50	50
S <sub>3</sub> P <sub>7</sub>	30	70
S <sub>0</sub> P <sub>10</sub>	0	100



**Fig. 1. Test cell for the compression test of model food powder. (a) Powder is in removable ring; (b) Excess powder is scraped off; (c) Powder is compressed.**



**Fig. 2.** Typical compression and decompression curves of cohesive and non-cohesive powders. A: Irrecoverable work; B: Recoverable work.

Irrecoverable work was determined based on the previous reports (Peleg *et al.*, 1982, Moreyra and Peleg, 1981). The force-deformation curves were recorded during compression-decompression tests (Fig. 2) by reversing the chart movement direction during decompression. The overall work was represented by the total area under the force deformation curve in compression and the irrecoverable work by the area bound by the hysteresis loop. The irreversible work was reported as follows. (%)

$$I_R (\%) = \frac{A}{A+B} \times (100) \quad (3)$$

where:  $I_R$  (%) = irrecoverable work

A = A area in Fig. 2

B = B area in Fig. 2

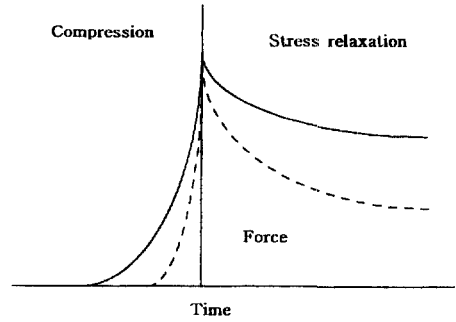
**Determination of stress relaxation patterns**

Powder specimen used for the compression tests was compressed with a 50 kg compression load cell of the Instron Universal Testing Machine (Model 1000) at a crosshead speed of 2.0 mm/min (Moreyra and Peleg, 1981, Peleg, 1979). After reaching 83% deformation (17% of the original volume) the crosshead was stopped and the force-relaxation curves (Fig. 3) were recorded at a chart speed of 20 mm/min.

The force relaxation curves were normalized and linearized by:

$$\frac{F_0 t}{F_0 - F(t)} = k_1 + k_2 t \quad (4)$$

where:  $k_1$  and  $k_2$  = constants characteristics of the actual shape of curve



**Fig. 3.** Typical deformation and relaxation curves of dry and wet powders. —: Dry powder, ---: Wet powder.

$F_0$  = initial force

$F(t)$  = force after time  $t$

$t$  = time (min)

While  $1/k_1$  depicts the initial relaxation rate,  $1/k_2$  is an asymptotic value of  $[F_0 - F(t)]/F_0$ . This enables calculation of an asymptotic modulus ( $E_A$ ) from:

$$E_A = \frac{F_0}{A\epsilon} (1 - 1/k_2) \quad (5)$$

where: A = cross sectional area of the specimen ( $cm^2$ )

$E_A$  = asymptotic modulus ( $kg/cm^2$ )

$\epsilon$  = strain

The magnitude of  $E_A$  can serve as a practical index of solidity. The strain can be calculated from:

$$\epsilon = \frac{\Delta H}{H_0} \quad (6)$$

where:  $H_0$  = height of specimen before deformation (mm)

$\Delta H$  = height of specimen after deformation (mm)

And % relaxation can be calculated from the following equation (13):

$$\% \text{ relaxation} = \frac{F(1 \text{ min})}{F_0} \times 100 \quad (7)$$

where:  $F(1 \text{ min})$  = forces recorded after 1 min at relaxation

**Statistical analysis**

Polynomial regressions were done using the Statistical Analysis System (SAS) (1985).

## Results and Discussion

### Compressibility, compaction ratio, and irrecoverable work

Compressibility values of model food powder are listed along with water activity levels in Table 2. As shown in the Table 2, compressibility of all the powder increased with increased water activity. This implies that even a non-cohesive powder may cause problem related to the decreased flowability when subjected to an environment of high humidity. As for the composition of a powder, the more proteinaceous the powder, the higher its compressibility. This means that, if a powder of high protein content is exposed to a high humidity environment, the powder may aggregate or solidify due to the increased compressibility.

Compaction ratios of model food powder are given in Table 3. Compaction ratios of all the powder increased with increase in water activity, implying the flowability of the powder can be decreased with increased water activity especially when the powder is kept under the pressure of 1 kg/cm<sup>2</sup>. In a humid environment powder shows higher compaction ratio and less flowability than in a dry environment. As for the powder composition the higher proteinaceous the powder, the

higher its compaction ratio. This means the proteinaceous powder may have more serious flow problem than the starchy powder.

The regression coefficients of quadratic model equations for compressibility and compaction ratios are also given in Table 2 and Table 3. In both Tables the coefficients of determination were so high (minimum 0.991 for compressibility and minimum 0.981 for compaction ratio) that those equations can be used successfully for the prediction of compression and compaction ratio of a powder at different humidity conditions.

The percent of irrecoverable work during compression and decompression of model food powder is given in Table 4. The low irrecoverable work implies that the powder returns easily to a free flowing state after removal of the external load. The irrecoverable work of all the powder increased with increased water activity. Especially, at the water activity of 0.76 or higher, the irrecoverable work was larger than 89%, implying the high possibility of producing agglomerate during powder processing at a humid condition. The more proteinaceous powder, the higher irrecoverable work. In other words, the proteinaceous powder is most likely to cause flow problems.

Table 4 also lists the regression coefficients of

**Table 2. Compressibility of model food powder and regression coefficients to predict compressibility at different water activity**

Model food powder	Compressibility								Regression coefficients				
	a <sub>w</sub>	0.12	0.23	0.33	0.44	0.53	0.65	0.76	0.84	a	b	c	R <sup>2</sup>
S <sub>10</sub> P <sub>0</sub>	0.006	0.007	0.008	0.010	0.012	0.015	0.018	0.021	0.025	0.0056	0.0010	0.0205	0.999
S <sub>10</sub> P <sub>3</sub>	0.008	0.009	0.010	0.012	0.015	0.019	0.021	0.024	0.024	0.0064	0.0040	0.0191	0.991
S <sub>5</sub> P <sub>5</sub>	0.010	0.010	0.012	0.013	0.015	0.019	0.024	0.027	0.027	0.0107	-0.001	0.0351	0.996
S <sub>3</sub> P <sub>7</sub>	0.012	0.013	0.014	0.016	0.019	0.023	0.026	0.029	0.029	0.0113	0.0015	0.0237	0.995
S <sub>0</sub> P <sub>10</sub>	0.013	0.014	0.014	0.016	0.020	0.024	0.028	0.031	0.031	0.0131	-0.006	0.0330	0.991

a,b,c are constants for the quadratic equation,  $y=a+bx+cx^2$ , where x=water activity, y=bulk flow properties

**Table 3. Compaction ratio of model food powder and regression coefficients to predict compaction ratio at different water activity**

Model food powder	Compaction ratio								Regression coefficients				
	a <sub>w</sub>	0.12	0.23	0.33	0.44	0.53	0.65	0.76	0.84	a	b	c	R <sup>2</sup>
S <sub>10</sub> P <sub>0</sub>	1.09	1.10	1.11	1.13	1.17	1.20	1.23	1.25	1.25	1.0748	0.0710	0.1719	0.988
S <sub>10</sub> P <sub>3</sub>	1.13	1.14	1.15	1.17	1.21	1.27	1.31	1.33	1.33	1.1211	0.0026	0.3101	0.984
S <sub>5</sub> P <sub>5</sub>	1.16	1.17	1.19	1.21	1.26	1.29	1.32	1.35	1.35	1.1380	0.1160	0.1656	0.988
S <sub>3</sub> P <sub>7</sub>	1.20	1.21	1.22	1.24	1.28	1.32	1.36	1.39	1.39	1.1935	-0.000	0.2851	0.994
S <sub>0</sub> P <sub>10</sub>	1.24	1.25	1.26	1.28	1.32	1.38	1.41	1.43	1.43	1.2271	0.0338	0.2638	0.981

a,b,c are constants for the quadratic equation,  $y=a+bx+cx^2$ , where x=water activity, y=bulk flow properties

quadratic equations for the irrecoverable work of model food powder. As shown in the table, the coefficients of determination was higher than 0.975. This suggests that the equations will be useful to predict the irrecoverable work of powder in an environment of different humidity.

#### Asymptotic modulus and relaxation (%)

Asymptotic modulus values decreased with increase in water activity levels (Table 5). As powder absorbs moisture at a humid condition, the powder may lose its solid, elastic properties. As for the powder composition the more proteinaceous the powder, the lower its asymptotic modulus values, implying a decreased flowability of the proteinaceous powder.

Relaxation (%) values indicating powder elasticity are given in Table 6. All the model food powder showed decrease in relaxation (%) as water activity levels increased. In general, the adsorbed water decreases the powder elasticity. As for the powder composition the more proteinaceous the powder, the lower its relaxation (%) values. In other words, the starchy powder retains the higher elasticity than the proteinaceous powder.

The regression coefficients of quadratic equation for asymptotic modulus and relaxation (%) are also shown in Table 5 and Table 6, respectively. The coefficients of determination were above 0.989 for asymptotic modulus and above 0.985 for relaxation (%), implying the reliability of the equations.

**Table 4. Irrecoverable work of model food powder and regression coefficients to predict irrecoverable work at different water activity**

Model food powder	Irrecoverable work (%)									Regression coefficients			
	a <sub>w</sub>	0.12	0.23	0.33	0.44	0.53	0.65	0.76	0.84	a	b	c	R <sup>2</sup>
S <sub>10</sub> P <sub>0</sub>	72	72	74	77	80	85	89	91	91	70.220	5.5807	24.628	0.990
S <sub>2</sub> P <sub>3</sub>	75	75	77	80	85	91	94	96	96	72.178	10.354	23.422	0.976
S <sub>3</sub> P <sub>5</sub>	76	77	79	81	84	92	96	97	97	74.519	4.5642	28.877	0.975
S <sub>3</sub> P <sub>7</sub>	79	80	82	85	89	93	97	98	98	76.154	15.531	14.086	0.986
S <sub>0</sub> P <sub>10</sub>	81	82	84	87	91	96	98	99	99	77.422	20.516	7.8998	0.976

\* a, b, c are constants for the quadratic equation,  $y=a+bx+cx^2$ , where x=water activity, y=bulk flow properties

**Table 5. Asymptotic modulus of model food powder and regression coefficients to predict asymptotic modulus at different water activity**

Model food powder	Asymptotic modulus (g/cm <sup>2</sup> )									Regression coefficients			
	a <sub>w</sub>	0.12	0.23	0.33	0.44	0.53	0.65	0.76	0.84	a	b	c	R <sup>2</sup>
S <sub>10</sub> P <sub>0</sub>	1.58	1.57	1.55	1.52	1.47	1.40	1.36	1.32	1.32	1.5978	-0.052	-0.344	0.991
S <sub>2</sub> P <sub>3</sub>	1.52	1.50	1.49	1.47	1.42	1.36	1.30	1.26	1.26	1.5187	0.0513	-0.437	0.994
S <sub>3</sub> P <sub>5</sub>	1.47	1.46	1.45	1.42	1.36	1.30	1.24	1.19	1.19	1.4730	0.0658	-0.488	0.994
S <sub>3</sub> P <sub>7</sub>	1.41	1.39	1.37	1.34	1.29	1.23	1.27	1.12	1.12	1.4189	-0.022	-0.402	0.998
S <sub>0</sub> P <sub>10</sub>	1.37	1.35	1.35	1.27	1.22	1.16	1.09	1.04	1.04	1.3954	-0.094	0.4014	0.989

a, b, c are constants for the quadratic equation,  $y=a+bx+cx^2$ , where x=water activity, y=bulk flow properties

**Table 6. Relaxation (%) of model food powder and regression coefficients to predict relaxation (%) at different water activity**

Model food powder	Relaxation (%)									Regression coefficients			
	a <sub>w</sub>	0.12	0.23	0.33	0.44	0.53	0.65	0.76	0.84	a	b	c	R <sup>2</sup>
S <sub>10</sub> P <sub>0</sub>	96.4	95.9	95.3	94.5	92.7	90.4	89.2	88.2	88.2	97.082	-2.859	-9.728	0.985
S <sub>2</sub> P <sub>3</sub>	95.6	95.1	94.6	93.8	92.5	90.9	88.4	87.5	87.5	95.600	1.4362	-13.58	0.995
S <sub>3</sub> P <sub>5</sub>	93.6	93.1	92.6	92.0	90.7	89.2	87.8	86.9	86.9	93.914	-1.242	-8.728	0.995
S <sub>3</sub> P <sub>7</sub>	90.3	89.9	89.4	88.9	87.7	86.2	85.4	84.8	84.8	90.847	-2.868	-5.514	0.986
S <sub>0</sub> P <sub>10</sub>	88.7	88.3	87.9	87.3	86.1	85.2	84.5	83.9	83.9	89.333	-3.790	3.3867	0.988

a, b, c are constants for the quadratic equation,  $y=a+bx+cx^2$ , where x=water activity, y=bulk flow properties

## Conclusions

It has been shown that the flowability-related physical properties of model food powder can be monitored by simple tests and can also be predicted by regressed model equations. The properties such as compressibility, compaction ratio, irrecoverable work, asymptotic modulus, and relaxation (%) are all quantifiable and provide numerical scales for powder quality or powder flow. Though the fitted equations to predict those properties are derived from empirical relationships, they are still meaningful because of their direct relation with the fundamental properties of a powder and the relative humidity of an environment.

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