
Sorption isotherms of flour and flow behaviour of dough as influenced by flour compaction

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Ramanathan, S. and S. Cenkowski. 1995. **Sorption isotherms of flour and flow behaviour of dough as influenced by flour compaction.** *Can. Agric. Eng.* 37:119-124. The influence of compaction of flour on its sorption isotherm and on the flow characteristics of a flour-water dough was investigated. The compacted flour equilibrated at nearly the same water activity (a_w) as the non-compacted flour in the whole range of the sorption isotherm. The flow parameters, namely the flow behaviour index and the consistency coefficient, were determined for the dough prepared using the non-compacted and compacted flour. The dough prepared using the compacted flour had a higher flow behaviour index and a lower consistency coefficient than that prepared using the non-compacted flour.

Keywords: equilibrium, water activity, rheology, flour, compaction.

Nous avons testé l'effet produit par la compression de la farine sur son isotherme de sorption et sur les caractéristiques d'écoulement d'une pâte faite d'eau et de farine. La farine comprimée s'est équilibrée au même état hygrométrique (water activity, a_w) - ou peu s'en faut - sur l'entière isotherme de sorption. Nous avons déterminé les paramètres d'écoulement pour les pâtes faites de farines comprimée et non-comprimée, c'est-à-dire, le coefficient de consistance et l'indice des variations des caractéristiques d'écoulement. La pâte faite de farine comprimée avait un indice d'écoulement plus élevé et un coefficient de consistance moindre que la pâte faite de farine non-comprimée.

INTRODUCTION

The importance of moisture sorption by flour has been established (Bailey 1920; Fairbrother 1929; Anker et al. 1942; Morey et al. 1947; Bushuk and Winkler 1957; Gur-Arieh et al. 1965a; Henderson and Pixton 1982). Bushuk and Winkler (1957) used the McBain-Bakr sorption balance in conjunction with the high vacuum technique to determine the adsorption and desorption isotherms of flour, starch, and gluten. They made the first ever attempt to account for the sorptive capacity of flour on the basis of its physical and chemical characteristics. Gur-Arieh et al. (1965a) obtained the sorption isotherms of flour by circulating air at various constant relative humidities in a closed system through a tube loosely packed with flour sample. To increase the precision of water activity determination for high moisture flour, Gur-Arieh et al. (1965b) also used the pressure membrane cell. Other researchers (Bailey 1920; Fairbrother 1929; Anker et al. 1942; Morey et al. 1947; Henderson and Pixton 1982) used the indirect method (constant-relative-humidity desiccator technique) to obtain the sorption isotherms of flour. In

this method, a sample of flour is allowed to equilibrate in a closed environment of constant relative humidity created using saturated salt solutions. Henderson and Pixton (1982) obtained the isotherms for wheat flour at 5, 15, and 25°C and found that the temperature had no effect on the sorptive capacity of the flour.

The influence of compaction of flour on its sorption isotherms has not been studied previously, although Bushuk and Winkler (1957) reported that the rate of moisture adsorption by flour was not affected by packing it more compactly. They did not attempt to find the water activity of flour as influenced by compaction. The experimental methodology for compaction was not explained in detail either.

The compaction of flour to 60% of its original volume can lead to a significant decrease in the storage volume and transportation cost. By compaction we mean the reduction of pore space and hence porosity. This in turn may prevent the diffusion of oxygen into the flour and thereby reduce enzymatic activity and infestations by mites and micro-organisms.

The importance of the rheological properties of the dough and the methods to evaluate them have been described (Hibberd and Parker 1975; Bushuk 1985; Faubion et al. 1985; Hosney 1985; Sharma et al. 1993). The quality of the finished loaf of bread depends on the rheological properties of the dough, which in turn depend on the flour quality. The flour quality might change during compaction and reconstitution stages which would reflect on the rheological properties of the dough made using that flour. Sharma et al. (1993) discussed in detail the importance of dough viscosity in production control and equipment design in automated bakeries and in assessing and controlling the quality of the baked product. They also discussed the importance of flow parameters of the dough, namely the flow behaviour index and the consistency coefficient, in the design and selection of suitable pumps for pumping the dough from the point of mixing to the fermentation chamber and then to the oven.

The main objectives of this research were to determine

- 1) the influence of compaction of flour on its moisture sorption isotherms, and
- 2) the changes in the flow parameters of the dough made using the flour reconstituted after compaction.

EXPERIMENTAL APPARATUS AND PROCEDURES

Experimental apparatus

a_w meter The apparatus used for the determination of water activity (a_w) of flour was the a_w meter (Novasina a_w Center, No:6114, Zurich, Switzerland). It was used to perform the a_w measurements at controlled temperatures. Here, the temperature equalization was accelerated by the forced admission or dissipation of heat.

The internal temperature of the a_w box could be digitally set to any value between 0 and 50°C (0.2°C). Ambient temperature changes would affect the internal temperature of the a_w box by a maximum of 0.04°C/°C.

Rheometer A capillary extrusion rheometer was used to obtain the flow behaviour index (n) and the consistency coefficient (K) of the dough prepared using the non-compacted and compacted flour (Sharma et al. 1993). The rheometer consisted of a brass cylinder having a threaded portion at one end (with an inside diameter of 19 mm, a thickness of 2.8 mm, and a height of 136 mm) and a hollow, brass capillary having threaded portions at both ends (with an inside diameter of 3 mm, and a height of 101 mm). The cylinder was screwed into the top portion and the capillary into the bottom portion of the supporting collar so that the bottom surface of the cylinder was flush with the top surface of the capillary. Also, the collar converged from the diameter of the cylinder to the diameter of the capillary at their point of contact. A close fitting brass plunger with a working length of 330 mm was used to push the dough through the capillary.

The brass plunger was connected to the load cell of a Universal Testing Machine (UTM - Model ET 1100, John Chatillon & Sons Inc., Kew Gardens, NY). The UTM machine was equipped with a 5 kN load cell. The supporting collar with the cylinder at the top and the capillary at the bottom was placed on a stand and the entire assembly was placed on the movable ram of the UTM. The complete unit was positioned under the crosshead of the UTM so that when the ram moved up, the plunger smoothly slid down inside the cylinder and thereby extruded the dough through the capillary.

Description and preparation of test samples

The flour used in this research was the all-purpose flour (Robin Hood, Multifoods Inc., Markham, ON) obtained from a local supplier. It had an initial moisture content of 11.8% dry basis (db) and a protein content of 12.6% on a 14% moisture absorption basis.

For the sorption equilibrium moisture tests, the moisture content of the flour was raised by circulating the water vapour through four or five thin layers (each having an approximate thickness of 3–4 mm) of flour with an air pump. The process of moisturization was done for different periods of time in order to get samples of different moisture contents. To obtain the samples of low moisture content, the flour was dried in a convection oven at 40°C for 1, 2, and 3 h. The temperature of 40°C was selected because Bushuk and Winkler (1957) found that the heat treatment of the flour at 100°C for 24 h reduced its sorptive capacity by 20%. At the end of each conditioning process, the sample was mixed

thoroughly in a plastic Ziploc bag and allowed to equilibrate for 24 h at a temperature of 10°C.

The compaction of flour for sorption tests was achieved in the form of small pellets of 6.5 mm diameter and 10 mm height using the cylinder-plunger arrangement. A hollow brass cylinder (6.5 mm inside diameter and 25 mm height) was filled with flour and placed on the movable ram of the UTM. A close fitting brass plunger, having the same working length as the 25 mm height of the cylinder, was connected to the load cell of the UTM. When the ram moved up, the plunger slid down inside the cylinder to compact the flour to 60% of its original volume. The pellets were obtained from the flours of different moisture contents prepared in the above manner and the a_w 's were determined. A similar arrangement was used for the compaction of flour for dough flow behaviour tests except that the cylinder had an inside diameter of 19 mm and a height of 300 mm. A known mass of flour (100 g) at 11.8% db moisture content was compacted to a known deformation of 60% of its original volume. The compacted samples were friable due to the low moisture content of the flour (11.8% db) and needed to be handled carefully.

The moisture content of the flour was determined according to the Approved Methods of the American Association of Cereal Chemists 44-15A (AACC 1983a) by drying 3 g of sample in a convection oven at 130 ± 1°C for 1 h.

Dough prepared from the flour was characterized by Farinograph and Mixograph mixing curves according to the Approved Methods of the AACC 54-21 (AACC 1983b) and 54-40A (AACC 1983c), respectively. Farinograph absorption was 63.5% with dough development time 8.0 min and a mixing tolerance index of 10 BU (Brabender Units). Mixograph peak time was 3.5 min and peak height 480 MU (Mixograph Units) at 64% absorption. Subsequently, larger quantities of dough were prepared by mixing 100 g of flour with 66 g of water for 3.5 min in a pin mixer (Hlynka and Anderson 1955). When the compacted flour was tested for Farinograph Absorption and Mixograph Mixing time, it was found that there was no significant change in either of the characteristics.

Experimental procedure

a_w meter operation Prior to the determination of a_w , the samples were held at room temperature, the same as the a_w meter, for 1 h. This allowed the samples to come to approximate thermal equilibrium with the temperature at which they were to be tested. The temperature in a control chamber where the meter was placed was adjusted to 25°C. It took approximately 60 min for the temperature equilibrium to be attained.

Approximately 7.5 g of flour was put into the sample bowl. The sample bowl was placed in the measuring bowl which in turn was positioned under the sensor inside the box. This allowed the system to heat or cool the sample and the measuring bowl to the selected temperature. According to the operation manual, the equilibrium was reached provided the a_w value was constant for 60 min. In our experiments, the waiting time was extended to 2 h for better accuracy. Once the equilibrium was reached, the a_w value was recorded and the moisture content of the flour was determined using the

method described in the previous section. The same procedure was repeated for the flours prepared to different moisture contents and for the compacted flour pellets.

Rheometer operation The cylinder, filled with dough without any air gaps, and the capillary were screwed on to the top and bottom of the supporting collar, respectively. The entire assembly was held on a stand and the unit was placed on a flat plate on the movable ram of the UTM. When the ram moved up, the plunger slid down inside the cylinder and force-extruded the dough through the capillary.

The force required to extrude the dough through the capillary was recorded using a data acquisition program run on a personal computer connected to the UTM through an RS-232 port. To determine the force drop in the capillary, the force at the point of entry of the dough into the capillary had to be measured. This was measured by removing the capillary and recording the force when the dough was about to extrude through the bottom of the supporting collar (having the same diameter as that of the capillary). The dough density was determined by the volume displacement method (volume of water displaced by a known mass of dough) (Mohsenin 1986). The experiment was repeated for different ram speeds. For each speed, the mass flow rate of the dough was calculated. The volumetric flow rate of the dough was calculated by dividing the mass flow rate of the dough by dough density. This also was calculated theoretically by multiplying the speed of the ram by the cross sectional area of the plunger. The above experiment was repeated for the dough prepared using the flour reconstituted after compaction.

Experimental design

The water activity of flour was measured at moisture content levels of 5, 8, 10.5, 11.8, 14, 16.5, 18, 20, and 24% db and at a temperature of 25°C. Three replicates made at each test level were used to determine the moisture sorption isotherms of flour. The same procedure was repeated in the case of compacted flour pellets.

In determining the flow parameters of the dough, the pressure drop and the flow rate values were measured for the ram speed of 5 mm/min. The experiments were repeated for speeds of 10, 20, 50, 100, 200, and 350 mm/min. Three replicates made at each ram speed were used to determine the flow parameters of the dough. The same procedure was repeated for the dough prepared using the compacted flour.

ANALYSIS OF DATA

The analysis of sorption data was based on fitting the data to the Chung and Pfof equation (Chung and Pfof 1967):

$$\ln(a_w) = \frac{A}{RT} \exp(-BM) \quad (1)$$

where:

- a_w = water activity (fraction),
- M = equilibrium moisture content (% db),
- T = temperature (K),
- R = universal gas constant (8.314 J•mol⁻¹K⁻¹), and
- A, B = constants.

A statistical analysis software (Proc NLIN, SAS 1985) was used to compare the constants A and B determined for

non-compacted flour to the constants in Eq. 1 determined for the flour brought to equilibrium after 60% compaction.

For the extrusion tests, the wall shear stress in a capillary was calculated as (Toledo 1991):

$$\tau = \frac{\Delta P r}{2L} \quad (2)$$

where:

- τ = shear stress (Pa),
- ΔP = pressure drop across the capillary (Pa),
- r = radius of the capillary (m), and
- L = length of the capillary (m).

The pressure drop in the capillary was determined as:

$$\Delta P = \frac{F_1 - F_2}{A} \quad (3)$$

where:

- F_1 = force at the point where the dough was about to extrude through the capillary (N),
- F_2 = force at the point of entry of the dough into the capillary (N). (This is the same as the force at the point where the dough would leave the cylinder because the bottom surface of the cylinder was flush with the top surface of the capillary), and
- A = area of cross section of the capillary (m²).

The apparent shear rate was calculated as (Toledo 1991):

$$\dot{\gamma} = \frac{4Q}{\pi r^3} \quad (4)$$

where:

- $\dot{\gamma}$ = shear rate (s⁻¹),
- Q = volumetric flow rate (m³•s⁻¹), and
- r = radius of the capillary (m).

The Rabinowitsch-Mooney correction (Toledo 1991) was applied to obtain the corrected shear rates data:

$$CF = \frac{3}{4} + \frac{1}{4n} \quad (5)$$

where:

- CF = correction factor, and
- n = slope of the plot of log shear stress and log shear rate.

The corrected shear rate was then obtained by multiplying the apparent shear rate by the CF :

$$\dot{\gamma}_w = \dot{\gamma} CF \quad (6)$$

where:

- $\dot{\gamma}_w$ = the corrected shear rate (s⁻¹).

Using the power law model (Toledo 1991), the flow characteristic was determined from the shear stress and shear rate relationship:

$$\tau = K \dot{\gamma}_w^n \quad (7)$$

where:

- n = flow behaviour index, and

K = consistency coefficient.

The effect of compaction of flour was quantified by determining the parameters n and K for the dough prepared from compacted and non-compacted flour. Proc NLIN program (SAS 1985) was used to estimate whether the two parameters were significantly different (at 95% confidence interval) for the dough prepared from compacted and non-compacted flour.

RESULTS AND DISCUSSION

To verify the method and the equipment used in our research, the sorption isotherm data of the present work were compared with those published by Gur-Arieh et al. (1965b) for hard wheat flour and with those published by Bushuk and Winkler (1957) for flour milled from high grade Canadian hard red spring wheat. The comparison is given in Fig. 1. There is good agreement in the sorption isotherm data up to 0.7 a_w beyond which our data differ from the published data. The reason for this could have been that all-purpose flour was used in this research. This flour contained some additives (e.g. oxidants like ascorbic acid and enzymes like amylase) which could have altered its sorptive capacity. Iglesias and

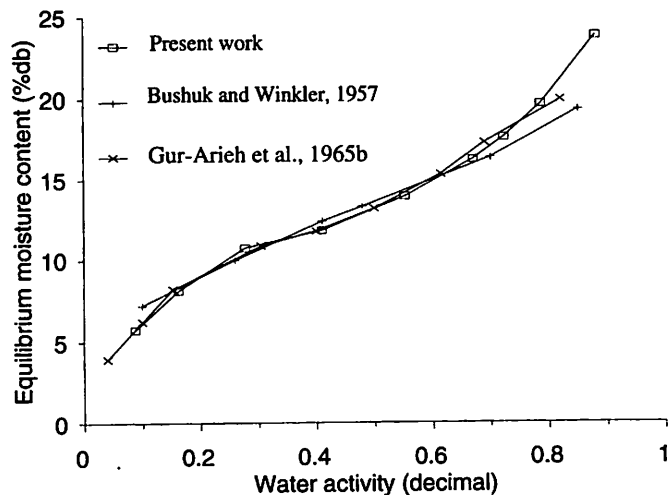


Fig. 1. A comparison of the present data with the published data.

Chirife (1975) showed that the moisture sorption curves of a material represent the integrated hygroscopic properties of its constituents, with each one being dominant in a particular range of water activity.

Figure 2 shows the comparison of sorption isotherms of non-compacted and compacted flour pellets at 25°C. Symbols represent the average values of three replicates and the horizontal bars indicate the 95% confidence limits. Lack of bars indicates that the size of symbols is in the range of the confidence bar size.

According to the classification of isotherms by Brunauer et al. (1940), the sorption isotherms for non-compacted and

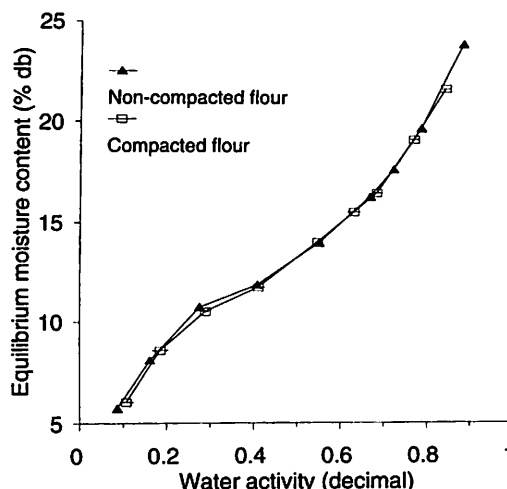


Fig. 2. Sorption isotherms of non-compacted and compacted flour at 25°C. Horizontal bars indicate confidence limits for a_w of compacted flour at 95% level.

compacted flour were of sigmoidal shape of Type II. For any given equilibrium moisture content, the compacted flour pellets equilibrated at a slightly higher a_w as compared to the non-compacted flour up to an a_w of 0.45 beyond which it was almost the same. However, statistically this difference was not significant. Proc NLIN (SAS 1985) was used to estimate the parameters of the two parameter model proposed by Chung and Pfof (1967), based on the sorption data of non-compacted flour and compacted flour pellets. The estimates of the parameters with their standard error and confidence intervals (95%) are given in Table I. The parameter estimates for non-compacted flour were not significantly different from those of compacted flour pellets. Thus, the sorption isotherm of flour at 25°C was not significantly influenced by compaction. Berry and Dickerson (1973) studied the moisture sorption isotherms of laying mash powder and laying mash pellets and concluded that at water activities up to 0.7, the pellets equilibrated at about 0.5 percent higher moisture content than did the powder which was in contrast with our findings.

Table I. Estimates of parameters of Chung and Pfof equation (Chung and Pfof 1967), with standard error and confidence intervals, for non-compacted and compacted flour equilibrated at 25°C

Material	Parameters in Eq. 1	Estimate	Std. error	95% Confidence interval	
				Lower	Upper
Non-compacted flour	A	18892	1990	14186	23598
	B	0.178	0.008	0.159	0.197
Compacted flour pellets	A	17982	1498	14441	21524
	B	0.176	0.006	0.161	0.191

Table II. Shear stress and shear rate values obtained at different crosshead speeds for the dough prepared using the non-compacted and compacted flour

Ram speed (mm/min)	Flow rate Q ($10^{-9} \text{ m}^3/\text{s}$)	Shear rate (s^{-1})	Non-compacted flour dough		Compacted flour dough	
			Force (N)	Shear stress (kPa)	Force (N)	Shear stress (kPa)
5	23.1	8.1	134	3.67	93	2.55
10	46.3	16.1	177	4.85	154	4.22
20	92.5	32.3	243	6.66	180	4.93
50	231.3	80.6	437	11.98	325	8.91
100	462.7	161.3	664	18.20	699	19.16
200	925.3	322.6	741	20.31	873	23.93
350	1619.3	564.5	1015	27.82	1179	32.31

the shear rate is non-linear, a flour-water dough is considered to follow the behaviour of a non-Newtonian fluid. A linear regression was performed using the values of log shear stress ($\log \tau$) as the dependent variable and log shear rate ($\log \dot{\gamma}$) as the independent variable (Toledo 1991). The slope of the regression represents the flow behaviour index (n) of the dough. The Rabinowitsch-Mooney correction factor (CF) was determined for the dough prepared using the non-compacted and compacted flour (Eq. 5). The corrected shear rate values were obtained by multiplying the apparent shear rate values by the CF (Eq. 6). Figure 3 shows a plot of shear stress against corrected shear rate. The shape of the plot for the dough prepared using the non-compacted and compacted flour is the same as that of a pseudo-plastic fluid

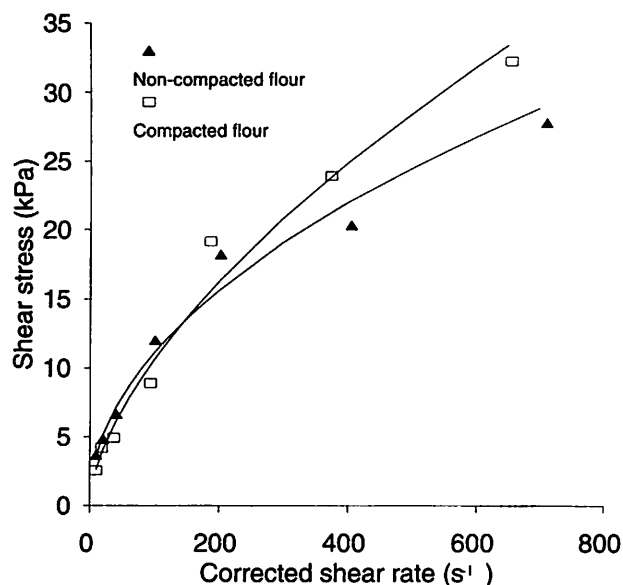


Fig. 3. Relationship between shear stress and corrected shear rate for the dough prepared using the non-compacted and compacted flour.

Table II gives the force and volumetric flow rate of the dough, calculated based on Eqs. 2 and 3, prepared using the non-compacted and compacted flour, at different ram speeds. At low ram speeds (up to 50 mm/min), the force required to extrude the dough prepared using the non-compacted flour was higher than that prepared with the compacted flour and vice versa at higher ram speeds. It was noticed that increasing ram speed increased both the shear rate and the pressure drop. The pressure drop for the water-flour dough was proportional to the volumetric flow rate of the dough. Since the relationship between the shear stress and

(Toledo 1991), indicating that the apparent viscosity of the dough decreases with increasing shear rate. These data of the shear stress and corrected shear rate were fitted to the power law model (Eq. 7) and Proc NLIN (SAS 1985) was used to estimate the parameters of the equation. Table III gives the parameters of the equation, with their standard error and confidence intervals, for the dough prepared using the non-compacted flour as control and the flour reconstituted after compaction. The dough prepared using the compacted flour had a 20% higher n value and a 45% lower consistency coefficient K than the dough prepared using non-compacted flour. The change in the physical parameters indicated that the compaction of flour changed its characteristics and hence the dough prepared using the compacted flour was less consistent with better flowability.

CONCLUSIONS

Based on the findings of this study, the following conclusions can be drawn.

- 1) The compaction of flour to 60% of its original volume had no influence on its moisture sorption isotherm in the a_w range between 0.09 and 0.88 and for the equilibration temperature 25°C.
- 2) The dough prepared using the non-compacted flour had

Table III. Flow parameters of dough with their standard error and confidence intervals

Material	Parameters in Eq. 7	Estimate	Std. error	95% Confidence interval	
				Lower	Upper
Non-compacted flour dough	K	1419	307	629	2209
	n	0.54	0.036	0.360	0.547
Compacted flour dough	K	770	242	149	1391
	n	0.580	0.052	0.446	0.714

a 20% higher flow behaviour index and a 45% lower consistency coefficient than that prepared using the non-compacted flour.

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