THE LIQUID PHASE OF DOUGH AND ITS ROLE IN BAKING

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ABSTRACT Cereal Chemistry 53(3): 318-326

The liquid phase of wheat-flour dough was separated by centrifugation at high gravitational fields. The water content of the remaining solid phase was 34.5%. Protein, hydrolysate lipid, water, and salt contents of the liquid phase were determined. Interchanging liquid phases of normal and defatted flour doughs for baking tests produced inconclusive results because of the establishment of new equilibria. Both electrical conductivity and gas retention of dough fell steeply with decreasing water content, extrapolating to zero at a water content near 34%, corresponding to the disappearance of the liquid phase. The foaming properties of liquid phases from doughs of differing lipid contents correlated with baked loaf volume when the baking test omitted an intermediate molding step. The inclusion or omission of the molding step caused large differences in baking performance for flours of higher lipid content but had little effect on flours of low lipid content. Rapid expansion of dough pieces caused by subjecting them to low pressure gave maximum volumes which correlated better than alveograms with baked loaf volume. The results point to the importance in baking of liquid lamellae surrounding gas bubbles in dough and the adsorbed films which stabilize them.

A number of independent physical methods have been used to estimate the amounts of "free" and "bound" water in wheat-flour doughs. These methods included freezing-point depression (1,2), N.M.R. (3), calorimetric determination of freezable water (4), equilibrium uptake of water by flour components in a humidity-controlled environment (5), extrapolation of farinograph measurements to zero mobility (6), and extrapolation of proteolytic enzyme activity to zero activity (7). Most methods have pointed to a critical moisture content of doughs of about 0.3 g/g dry flour, above which "free" water exists, although there has been some variability and a value as high as 0.7 g/g dry flour has been suggested (6). These results indicate that a flour-water system exists as one phase below this critical water content, and at higher water contents a second liquid phase is present.

The presence of a liquid phase has been confirmed by centrifuging doughs in very high gravitational fields. Baker et al. (8) discussed the properties and compositions of liquid phases centrifuged from doughs made from 25 different flours. Mauritzen and Stewart (9,10) centrifuged doughs at high speeds and carried out detailed analyses of the layers produced, including the dough liquor.

Baker et al. (8) showed that by using dough liquor to furnish the required water in breadmaking, beneficial effects on loaf volume and crust color were obtained. They rightly pointed out that the liquid phase of dough provides the medium through which reactions occur during manipulation and fermentation. It seems reasonable to believe that the liquid phase would also be the medium for the formation (during mixing) and subsequent growth (during fermentation) of gas bubbles in dough. The large effects on loaf volume of small amounts of lipid added to defatted flour (11) suggested that these compounds might exert their effects by modifying the properties of adsorbed films formed at the air/water interface. The nature of these mixed films of protein and lipid would then

determine the stability of thin liquid lamellae surrounding the gas bubbles of dough in much the same way as foam stability is determined in purely liquid media.

The present work was directed toward investigating the liquid phase in more detail and trying to assess its importance in the baking process.

MATERIALS AND METHODS

The flour used in this study was milled from the Australian prime hard wheat variety, Timgalen. It had a protein content of 13.4% (N × 5.7) and hydrolysate lipid contents of 1.44 and 0.38% before and after defatting (14% m.b.).

Doughs were centrifuged in an MSE Superspeed 65 preparative ultracentrifuge, in which the maximum field attainable was 407,000 × g.

Electrical conductivity was measured with a Radiometer conductivity meter type CDM2d using an electrode type CDC2114b. This electrode was calibrated against a standard conductivity cell using potassium chloride solutions of different known concentrations.

Foam stability was measured using a Pyrex filter (No. 3760) in which nitrogen gas was blown through a sintered glass disk into the liquid. The rate of flow was measured with a flowmeter and fixed at 15 cm^3/min. Next, 7 cm^3 of liquid was placed in the filter and the stopwatch was started when the foam column had reached a height of 7 cm. The time required for the column to fall to half its original height ($T_{1/2}$) was used as a relative measure of foam stability. Reported values of $T_{1/2}$ are means of triplicate determinations. The average standard deviation, expressed as a percentage of the mean, was 7%.

The method of extraction of lipid, hydrolysate lipid analyses, and the baking test procedure were the same as described previously (11), with the exception of the following modifications. In the baking test, the weight (wt) of the dough piece was measured immediately prior to tinning and the measured loaf volume adjusted by multiplying it by the factor 55/wt. This eliminated errors due to random losses of small pieces of dough in the molder, and it improved reproducibility. A previous experiment had established that loaf volume and weight of the dough piece were linearly related for dough weights between 50 and 60 g. When lipid was added back to defatted flour, it was first dissolved in chloroform and the flour slurried with the solution. The chloroform was then allowed to evaporate. This entirely eliminated the lack of reproducibility observed previously (11) when no predispersion of the lipid was attempted. Chloroform treatment of flour has been shown previously to have no effect on loaf volume (11).

The method for obtaining Chopin Alveograms was that of the ICC Standard No. 21.

Fermentograms were measured using an S.J.A. Fermentation Recorder Type 450, and the procedure outlined in the instruction manual for this instrument was followed.

For the dough expansion test at low pressure, the same size dough sample used in the baking test (30.2 g dry flour) was mixed to peak consistency and placed in a desiccator which was rapidly evacuated. Dough pieces expanded, reaching a maximum height about 30 sec after commencing evacuation. Heights reached by dough pieces were measured on a vertical scale which was placed inside the

desiccator; these heights were converted to volumes using a previously determined conversion graph. Values reported are means of triplicate determinations. The average standard deviation for this measurement was 9 cm^3.

Protein was analyzed by the Kjeldahl method. Sodium chloride was determined by a Mohr titration, using potassium chromate as indicator. Dry weights of the solid phases of dough were estimated by thoroughly removing the liquid supernatant, including the underlying gelatinous layer, and heating representative samples of about 1 g at 120°C to constant weight (1 hr). Values plotted are means of triplicate determinations, the standard deviation for the water content being 0.5%. Doughs, other than those used in baking tests, were made by addition of water and sodium chloride (2 g/100 g flour) to the flour.

RESULTS

Separation of Liquid Phase by Centrifugation

Doughs of 46% water content were mixed to peak consistency in a mixograph and centrifuged for 50 min at various speeds. The water content of the solid phase is plotted against centrifugal field in Fig. 1. Above about 100,000 × g, a limiting value is found for the amount of liquid phase which can be separated, corresponding to a solid-phase water content of 34.5%. The east of separation of the liquid phase depended on the degree of mixing. Doughs were mixed for various times, then spun at 25,000 × g for 50 min. The water content of the solid phase increased (*i.e.*, the rate of separation of the liquid phase was lower) as the
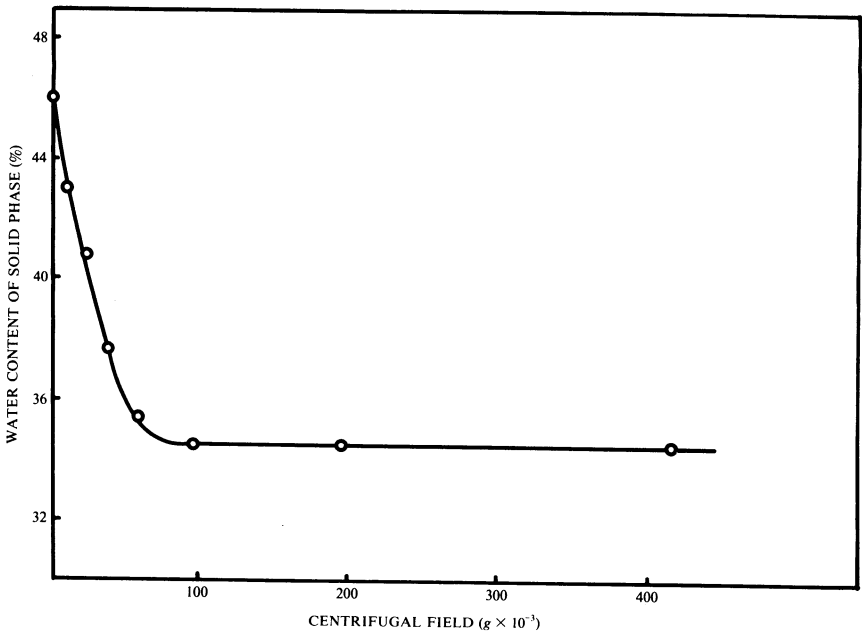

Fig. 1. Water content of solid phase of dough after centrifugation for 50 min at different values of the centrifugal field.

mixing time was increased to peak consistency, thereafter falling slightly. The limiting volume of liquid phase separated above $100,000 \times g$ was not significantly affected by the degree of mixing.

Composition of Liquid Phase

Table I shows some analytical data for the amounts and compositions of the liquid and solid phases of dough after separation by centrifugation. The gelatinous layer was not included in either of the two phases. Although analysis by the hydrolysate lipid method showed that a small but significant amount of lipid was present, no lipid could be extracted from the freeze-dried residue of the liquid phase using chloroform, reflecting strong binding of lipid to protein (12).

Effects of Quantity of Liquid Phase on Dough Conductivity and Loaf Volume

The conductivity of doughs of different water contents is shown in Fig. 2a. Doughs were mixed to peak consistency and allowed to relax for 15 min at 20°C before measurement. The conductivity of the liquid phase of dough (44% water content) was 40 millimhos/cm. As the water content is reduced, the conductivity falls, the steep part of the curve extrapolating to zero near a water content of 34%.

The sensitivity of loaf volume to the water content of a dough is shown in Fig. 2b. Maximum volumes of dough pieces expanded at low pressure (see later section) are also plotted. For a water content of about 44%, loaf volume is at a maximum. As the water content is reduced, the loaf volume falls rapidly until, at a value of about 35%, there is negligible retention of gas by the dough.

TABLE I
Amounts and Compositions of Dough and Its Solid and Liquid Phases

	Dough		Solid Phase		Liquid Phase		
	wt g	% by wt of dough	wt g	% by wt of solid phase	wt g	% by wt of liquid phase	Amounts Not Recovered g
Total amount	58.0		47.0		11.0		...
Water	26.7	46.0	16.2	34.5	9.50	86.0	1.0
Protein	4.6	7.9	4.2	8.9	0.37	3.4	0.03
Hydrolysate lipid	0.50	0.9	0.47	1.0	0.03	0.3	...
Sodium chloride	0.70	1.2	0.37	0.8	0.33	3.0	...
Remainder	25.5 (mainly starch)	44	25.8 (mainly starch)	55	0.77 (mainly soluble carbohydrate)	7.0	

Fermentograph measurements showed that yeast activity fell with decreasing water content. For example, the gas production at a water content of 37.1% was 63% of that at 44.5%.

Foaming Properties of Liquid Phases

The liquid phases of doughs made from whole and defatted flours were separated and their foam stabilities measured. The dough liquor from defatted flour had exceptionally high foam stability ($T_{1/2} > 2$ hr) while that from whole flour had low stability ($T_{1/2} \approx 12$ min). These values correlated well with the volumes of loaves baked from defatted and whole flours, which were 203 and 138 cc, respectively, provided the baking test was carried out with the omission of the remolding step (after 20 min of fermentation). The liquid phases of whole and defatted flours were interchanged with the solid phases, yeast and improver were added, and the doughs were remixed, proofed, and baked. Loaf volumes were apparently determined by the origin of the solid phase in the dough, *i.e.*, the loaf made from the solid phase of defatted flour dough gave a volume of 200 cc and that made from the solid phase of whole flour dough gave a volume of 138 cc. When the doughs, in which liquid phases had been interchanged, were remixed and the doughs again centrifuged to separate the liquid phases, it was found that the foam stabilities, as well as the loaf volumes, depended only on the nature of
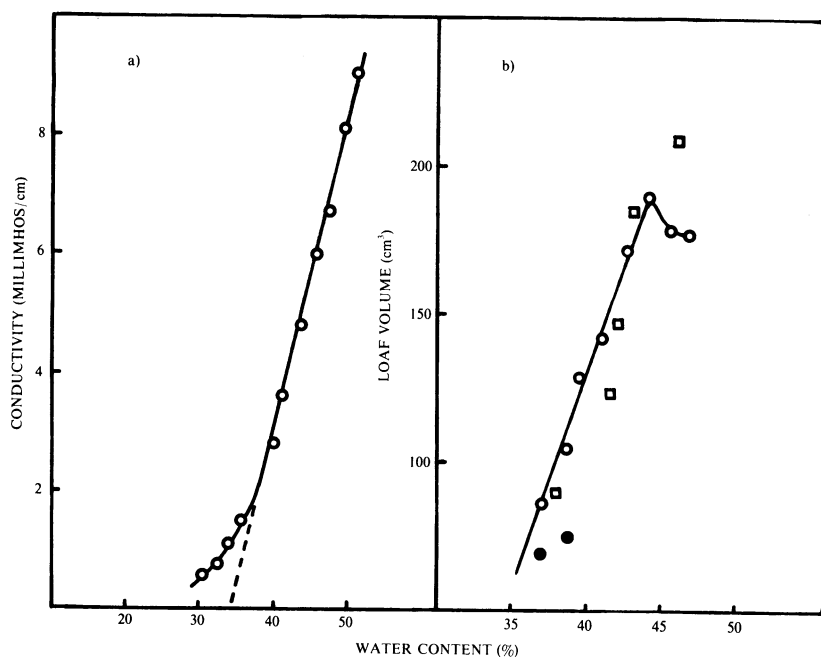

Fig. 2. a) Electrical conductivity of dough at 20°C as a function of water content. b) Loaf volume as a function of water content of dough; open circles, yeasted dough; closed circles, unyeasted dough; open squares, maximum volumes of dough pieces expanded at low pressure.

the solid phases. Thus, the measured values of $T_{1/2}$ were 2 hr and 12 min for doughs made with the solid phases of defatted and whole flours, respectively.

In Fig. 3a, loaf volume is plotted as a function of lipid content for two baking tests, one in which the remolding step was included and one in which it was omitted. Gas-cell structures were compared before and after the remolding step by freezing the dough pieces and subsequently examining them in section under the microscope. Dough pieces which were not remolded showed a very uneven gas-cell size distribution in which a few large holes were interspersed by many small cells. It was observed that at the end of the proofing stage, the large holes near the surface grew, usually forming large bubbles which were unstable and burst. Dough pieces which had been remolded showed a more uniform size distribution, the individual cells being of intermediate size and better separated.

In Fig. 3b the foam half-life ($T_{1/2}$) of the liquid phase is plotted as a function of lipid content of the flour. In the lower curve, the supernatants from the centrifuge tubes were combined without any precautions to avoid transfer of undissolved lipid. For the upper curve, the supernatants, after combination, were recentrifuged for 10 min at $10,000 \times g$ so as to form a lipid layer on the top. The liquid phase was then transferred to the foaming vessel by pipet, care being taken to avoid transfer of the insoluble lipid layer. If a small drop of flour lipid were
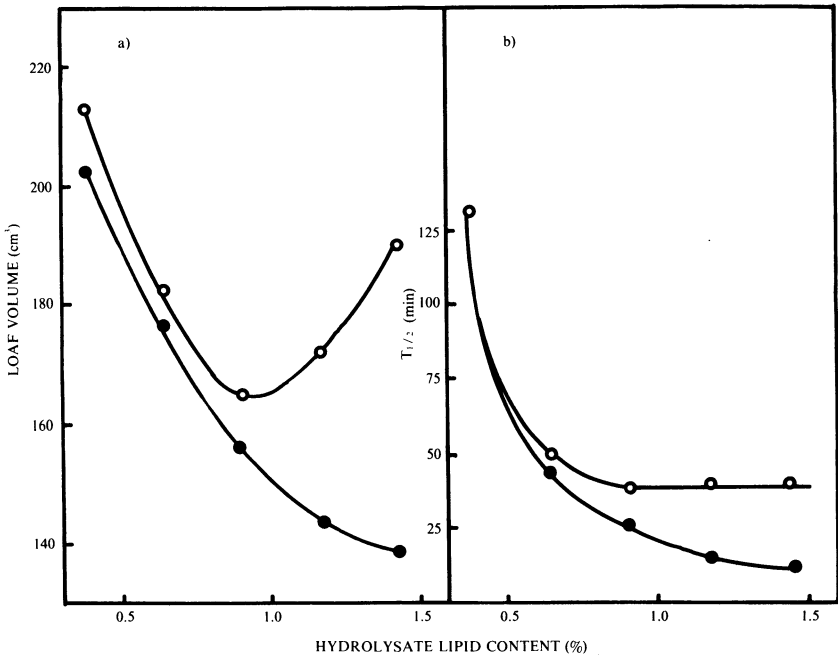

Fig. 3. a) Loaf volume as a function of hydrolysate lipid content of flour; open circles, remolding step included; closed circles, remolding step omitted. b) Foam half-life ($T_{1/2}$) of liquid phase of dough as a function of hydrolysate lipid content of flour; open circles, precautions taken to remove insoluble lipid; closed circles, no precautions taken to remove insoluble lipid.

touched against the top of a foam column prepared from the liquid phase of defatted flour, the foam, which is normally stable for more than an hour, broke down in a few minutes.

Expansion of Dough Pieces at Low Pressure

Dough pieces, placed in a rapidly evacuated desiccator, expanded to characteristic maximum sizes. A comparison between dough pieces made from whole and defatted flours is given in Table II. Details of loaf volumes from the baking test (no remold) and alveograms are included.

DISCUSSION

The constant value for the water content of the solid phase of dough above centrifugal fields of 100,000 × g (Fig. 1) suggests that most of the liquid phase has been separated. This value of 34.5% (approximately 0.5 g water/g solid) is higher than most values reported for the "bound" water content of doughs (3,4), which have been close to 0.3 g/g. This could be explained by the presence of a certain amount of mobile or "free" water in the solid phase which is therefore not removed by centrifugation. The solid phase is not strictly one phase, but consists of regions where the water content varies. For example, the water concentration associated with protein and insoluble pentosans is different and higher than that in the starch, and there may be mobile water associated with these components. Despite agreement between several workers on the figure of 0.3 g/g, a value of 0.38 g/g has been found recently for the water-binding capacity of wheat starch using differential scanning calorimetry (13).

The quantity and composition of the liquid phase agree with results of previous workers (8,9,10). Only about half of the solids content of the liquid phase is accounted for in Table I. From the results of Mauritzen and Stewart (9,10), soluble carbohydrates (mainly sugars) would be expected to make up most of the remainder.

To evaluate the role of a given component in a series of flours, the usual procedure is to separate the component, interchange it in the different flours, and observe changes in performance. The liquid phase is relatively easy to separate. However, on interchanging the liquid phases in different flour doughs, the compositions do not remain the same, and new equilibria are created. The composition of the liquid phase in a particular dough results from an equilibrium

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TABLE II
Comparison of Data for Baking Test, Alveograph, and
Expansion Test at Low Pressure for Whole and Defatted Flours

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Flour	Baking Test (No Remolding) Loaf vol cc	Alveograph		Expansion Test at Low Pressure Max vol cc
		Tenacity	Length	
Defatted	203	138	65	295
Whole	138	73	95	205

between solutes present in the liquid phase and the corresponding solid phase. This may explain the results obtained when liquid phases are interchanged between whole and defatted flour doughs.

At a water content of about 35%, there is effectively no gas retention by the dough. However, as the water content is increased, the loaf volume increases approximately linearly up to a water content of 44% (Fig. 2b). Webb *et al.* (14) found a similar critical value of the water content of doughs (~35%) by extrapolation of extensigraph extensibility-water content relations to zero extensibility. Although yeast activity falls with decreasing water content (7), fermentograph measurements showed that gas production does not limit the size of the loaf. The maximum volumes found in the expansion test at low pressure follow the same pattern as the baked loaf volumes, showing that gas retention is the limiting factor.

Two conclusions may be drawn from the electrical conductivity data. First, the steeply rising part of the conductivity-water content curve extrapolates to zero conductivity at a water content of about 34%, coincident with the disappearance of the liquid phase. The conductivity appears to be roughly proportional to the volume ratio of the liquid phase. Second, the liquid phase must be continuous in the dough although, judging by the variation in the ease of separation with mixing time, it may be concluded that the state of dispersion changes with degree of mixing. Small changes in conductivity of doughs with degree of mixing have been reported previously (15).

Figure 3 shows that there is good correlation between foam stability and loaf volume, provided the loaf volumes are compared in a baking test in which the remolding step after 20 min of fermentation is omitted. Foams formed from the liquid phase of defatted flour were extremely fine in bubble size and very stable. Loaves baked from defatted flour were similarly of very fine texture and high loaf volume, as reported previously (11). As the lipid content of the flour was increased, both the foam texture and stability and the loaf texture and volume progressively decreased (Fig. 3). The lipid complex of flour behaves essentially as an anti-foaming agent. It is not necessary for the lipid to be in solution in order to adsorb at the air/water interface. The undissolved lipid is able to spread at the interface, inducing instability in the liquid lamellae surrounding gas bubbles.

Although the decrease of loaf volume observed in the lower curve of Fig. 3a is apparently related to the foam destabilizing effects of increasing quantities of lipid, the dramatic effect of the remolding step on loaf volumes at higher lipid contents (upper curve) is not easily explained. The beneficial effects of punching and molding are well known and the resulting changes in the gas-cell structure have been reported previously (16). Possibly, the improvements effected in the gas-cell structure are more critical when films of poor stabilizing properties are adsorbed at the air/water interface. They would not be so important in flour doughs of low lipid content where the gas bubbles would have a high stability to rupture on coming into contact during expansion of the loaf.

Expansion of dough pieces by subjecting them to low pressure gives volumes which correlate well with loaf volumes measured in a baking test. By contrast, alveograms do not correlate well, the whole flour showing more balanced properties and having the greater extensibility, yet performing poorly in baking. It has been suggested that one reason why alveograms do not correlate well with baking performance is that a rate of deformation is used which is approximately

1000 times more rapid than that encountered in baking (17,18). However, the expansion test at low pressure employs a similar rate of expansion to the alveograph. The alveograph measures the capacity of a dough to be stretched into a very thin sheet. This is a most essential property for production of a good loaf volume but, for a dough which is mixed to optimum consistency, it may not usually be the limiting factor. The limit to gas retention capacity would be determined by the stability to rupture of the thin liquid lamellae which envelope the gas cells.

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