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EVALUATION OF SELECTED PROPERTIES OF CHLORINATED WHEAT FLOURS IN A LEAN CAKE FORMULATION

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Abstract

Cake flours treated with different levels of chlorine were evaluated by use in a Kissell cake formulation. Flour particle size distributions varied with chlorination level, but pH and specific gravity of the batters did not differ. Batter viscosity differences were observed at specific temperatures during heating of total batters. Temperature profiles differed positionally in the cakes, but these patterns of heat penetration were not related to level of chlorine treatment. However, water loss rates differed depending on the level of chlorine treatment indicating a more pronounced effect of chlorination level on the water loss characteristics of the cakes during the baking process than on the temperature profiles. The largest cake volume, contour deviations, and least shrinkage from the pan sides occurred with a 0.93 g Cl₂/kg flour treatment (commercial level). The SEM micrographs on crumb showed larger starch particles with more continuous and extensive matrix development between starch particles as chlorine level increased. The aforementioned characteristics prior, during, and after baking were related to factors contributing to optimal cake structure.

Introduction

Chlorine treatment of soft wheat flours has long been used to improve the baking performance of the flours used in cake making. Many researchers have studied the effects of chlorination on flour components and the functionality of flour in cake batter systems. Whistler et al. (1966) proposed a mechanism of starch depolymerization to explain the action of chlorine. Further work by Whistler and Pyler (1968) attempted to explain the action of chlorine on the polysaccharides found in flour. The work of Sollars (1961) and Gilles et al. (1964) was focused on determining the chlorine content of various flour fractions. Other workers have tried to evaluate changes in flour functionality that may be attributed to chlorine treatment. Youngquist et al. (1969) attributed the improving effect to an interaction between the chlorine and the lipids within the starch granules. Kissell et al. (1979) compared the functionality of lipids from untreated and chlorinated flours in white layer cake. Kulp et al. (1972) evaluated the changes in pasting characteristics, swelling power, extensibility, and water-binding capacity of the starch component after chlorination. Allen (1977) performed similar experiments but also included a study of the effect of chlorination on the heats of starch phase transitions. The changes in the functionality of the flour protein and subsequent effect on dough properties were examined by Tsen et al. (1971). Huang et al. (1982 a, b) examined the absorption of chlorine in starch and protein and found that the absorption of chlorine was a function of level of chlorination in protein but not starch. In isolated starch fractions, the response to chlorination depended on the stage of the phase transitions. For example, the initiation of the phase transition as measured by differential scanning calorimetry (DSC) was not influenced by level of chlorination, but swelling power after 80°C, and loss of birefringence at 90°C were affected. These studies have made major contributions to understanding the role of chlorine in individual components of the flours, although its precise action in baked products is not fully understood.

In this study, the functionality of the total cake flour which has been treated with different amounts of chlorine was evaluated in a research

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cake formulation. Temperature profiles and water loss rates were recorded throughout the baking process as had been done in previous studies by Gordon et al. (1979) and Hsu et al. (1980). Also, various batter and cake characteristics such as pH, viscosity, specific gravity, volume index and particle size distribution were determined. Scanning electron microscopy (SEM) examination and chlorine analysis by X-ray microanalysis were also included. The combination of studies of water loss rates and temperature profiles with macro- and microstructural evaluation should lead to a better understanding of the specific role that chlorine plays in flour functionality.

Materials and Methods

Batter Formulation and Baking Procedure

A modified Kissell cake formulation as described by Gordon et al. (1979) was used to prepare the cake batters. All ingredients except the flour were purchased in the retail market. The flour, with an ash content of 0.28% and protein content of 7.31% on a dry weight basis, was prepared by General Mills, Inc. at the following levels of chlorine treatment (g Cl₂/kg flour): 0 g/kg (pH 5.17); 0.31 g/kg (pH 5.09); 0.62 g/kg (pH 4.86); 0.93 g/kg (pH 4.51); and 1.24 g/kg (pH 4.48).

The cakes were baked in a controlled environment oven (Godsalve et al., 1977) at 190°C ± 1°C with an airflow rate of 10.1 m³/hr for 25 min. The temperature profiles were monitored by four thermocouples placed 5 mm above the bottom of the pan at the following positions: the center, 2.5, 5.1, and 6.9 cm radially from the center of the pan. The water loss rates from the cakes were calculated by monitoring the wet-bulb and dry-bulb temperatures of the airstreams flowing in and out of the oven throughout the baking process. These procedures were the same as those used by Gordon et al. (1979) for high starch cakes.

Flour Particle Size Analysis and Chlorine X-Ray Microanalysis in the SEM

The aluminum stubs used for particle size analysis were first coated with carbon paint, then covered with a thin layer of flour particles. The mounted samples were coated with carbon to minimize charging. These samples were then viewed in a Philips Model PSEM 500 scanning electron microscope operated at 6 kV. To ensure random selection of particles, measurements were taken at equidistant points along two perpendicular lines passing through the center of the stub. The areas of the particles were approximated by tracing the images of the particles as they appeared on the viewing screen of the microscope. The areas of these tracings were measured with a Hewlett Packard digitizer.

Chlorine analysis was performed on the flour particle components using energy dispersive X-ray microanalysis. The preparation of the sample stubs was similar to that described for particle size determinations. However, the samples were placed in a JEOL Model JSM-35 scanning electron microscope equipped with an Edax Model 711 microanalyzer. Random selection was accomplished by the same method described for particle size analysis. The X-ray intensity data were collected from the

samples for 400 sec time periods. The method of Nasir (1976) was used to calculate mass concentration ratios of minerals in the samples.

For the collection of X-ray data, the electron beam of the SEM was focused on various micro-components within the clumps of dry flour particles mounted on carbon-coated SEM stubs. Analysis of the lipid fractions was not included because of the high risk of interference from other components due to the nature of the lipid fraction and the size of the beam penetration. The determinations were done for the most part on the granular starch and wedge-type protein. Due to the nature of the starch and protein fractions, potassium (K) was used as the basis for the calculation of mass concentration ratios in starch fractions; sulfur (S), for protein fractions.

Determinations Made on the Batter

The pH of the complete batter was measured on a Corning Model 7 pH meter. A Fischer-Grease pycnometer was used to determine the specific gravity of the batter. A preliminary study of viscosity was performed on a flour-water slurry (1:9) in a Model AV-30 Brabender amylograph viscosograph. Flour (50 g) and distilled water (450 g) were initially mixed and used with a 350 cm-g cartridge at a rotational speed of 75 RPM for 42.5 min at a rate of temperature change of 1.5°C per min (0 time is 30°C). The viscosity of the batter was determined in a Model RV2 Haake rotoviscometer with an MV11 cylinder set. The unit was operated at 2 RPM in the temperature range 75-95°C. Readings were taken at 5, 10 and 15 min.

Measurement of the Final Cake Characteristics

Cross-sectional areas of the bisected cakes recorded using a Hewlett Packard digitizer (average of three tracings) were used as a volume indicator. The diameter of the cross-section was used as an index of shrinkage. Contour index was defined as the differences between center height and the average of the two side heights of the cross-sectional tracings.

Scanning Electron Microscopy Evaluation of Cake Crumb

Samples to be viewed by SEM were taken from cakes at four positions: the center bottom, the center top, mid-point (3.5 cm from the center), and the mid-point of the outer edge of the cake. Using a razor blade, the samples of crumb were cut as elongated triangles (4 mm x 5 mm) which were 1 mm thick. After transferring the crumb onto an aluminum stub coated with a thin layer of silver paint, the sample was placed in a desiccator and exposed overnight to osmium vapors. The fixed samples were then coated with palladium gold prior to viewing in a Philips Model PSEM 500 scanning electron microscope operated at 6 kV or 12 kV.

Results

Flour Particle Size Analysis and Chlorine Content

Figure 1 shows the size distributions of flour particles for 0 g/kg and 1.24 g/kg level of chlorine treatment. The distribution suggests that chlorination increases the clumping tendency of the flour as evidenced by larger areas recorded for the 1.24 g/kg flour. Furthermore, a bimodal distribution is evident for the 0 g/kg level. Looking at the 0 g/kg sample, only 49% of the par-

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ticles measured fell into the size range greater than $10 \times 10^{-5} \text{ cm}^2$, while in the 1.24 g/kg sample, 72% were in this size range. The increased clustering with increased chlorination is also evident in the intermediate levels of treatment. It is difficult to determine if the increased association of the flour particles observed in the dry state is maintained during the batter preparation and cake baking. Although Seguchi and Matsuki (1977) found marked clustering of chlorinated samples in suspension, the clumping tendency may already have been established in the dry form. In Fig. 2 we see that the chlorine is taken up by starch more gradually than in the protein fraction of the same flour samples. Sollars (1961) found little chlorine located in the extracted starch fraction, although starch functionality seemed to be greatly affected after chlorine treatment. Gilles et al. (1964) confirmed that little chlorine is associated with the starch. After they made corrections for the lipids on the starch granules, the starch fraction showed no consistent increase with increased chlorine treatment. However, they found a slight increase in the chlorine content for the gluten fraction. In the present study, the correction for lipids was not made, possibly explaining the slight increase in chlorine uptake observed in the starch components in Fig. 2. We see, then, that the flour particle sizes are somewhat larger in the chlorine treated samples and that the chlorine uptake is somewhat greater for protein than for starch.

Batter Characteristics

It was found that the pH of the batters did not vary greatly from one level of chlorine to the next (Table 1). The batters were at 7.3-7.4 pH at all times. The pH of flour-water slurries decreased as the level of chlorine treatment was increased (pH 5.17 for 0 level and 4.48 for 1.24), but the pH of the batter was not affected by these initial differences. Localized areas may be affected by a lowering of pH, but this could not be detected in our batter pH measurements. The values for specific gravity of the batters were within the range of 0.98-0.99 regardless of the level of chlorine treatment.

A preliminary viscosity study of flour-water slurries on the Brabender amylograph (Fig. 3) showed that as the level of chlorine treatment was increased to 0.93 g/kg, the maximum viscosity of the flour-water slurries increased, but at the 1.24 g/kg level, the viscosity was lower than that of the 0.93 g/kg level. Unpublished data by B.M. Dirks did not have similar differences in peak viscosity. The reason for the differences found by those researchers is not obvious. Allen (1977) showed that the differential effects of chlorine on viscosity as measured by the Brabender amylograph are dependent on starch concentration. Therefore, the Haake rotoviscometer was used for batters so that determinations could be made on the undiluted batters. Table 2 shows the viscosities that are recorded over a range of temperatures for the batters prepared from untreated and commercially treated flours. The differences between the batter systems were not as dramatic as the results obtained for the flour-water slurries

done by the Brabender amylograph method. The increase in viscosity is not measurable until the temperatures approach the gelatinization temperatures of the starch in high sucrose batter

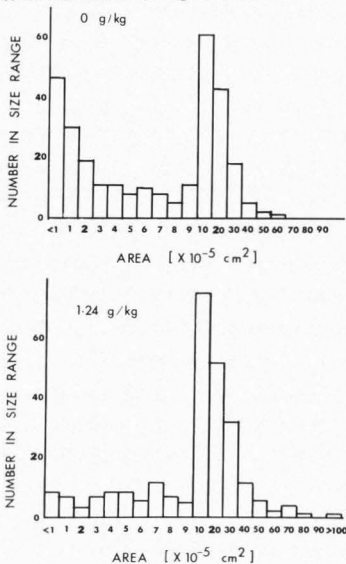


Fig. 1 Size distributions of flour particles for 0 and 1.24 g Cl_2/kg flour treatment.

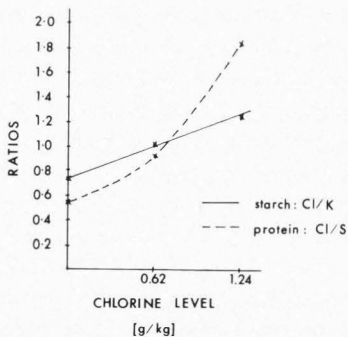


Fig. 2 Relative amounts of chlorine taken up by starch (Cl/K) and protein (Cl/S) for 3 different levels of chlorine treated flour.

Table 1. Summary of physical measurements on batters and final cakes.

Measurement	Level of chlorine g/kg				
	0	0.31	0.62	0.93	1.24
pH batter	7.4	7.4	7.3	7.4	7.4
Specific gravity (batter)	0.98	0.98	0.99	0.99	0.98
Cake cross-sectional area (cm ²)	22.12	24.45	28.64	41.02	39.47
Cake contour index	-0.28	-0.025	0.18	0.81	0.60
Cake diameter (cm)	13.1	13.6	13.6	14.3	13.9

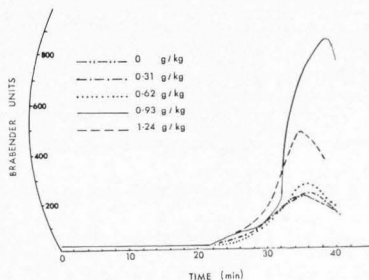


Fig. 3 Brabender viscosity curves for flour-water slurries.

systems. Slight differences between the samples were observed when viscosities are measured at 85°C. There is a suggestion that the increase in viscosity as temperature increases is more rapid in the chlorinated samples. This trend is also evident when the batters are held at 90°C. It appears that the level of chlorine treatment (up to 0.93 g/kg) does not dramatically influence the final viscosity of the batter, but that the rate of viscosity development may be more rapid due to chlorination. Although other workers have emphasized the importance of batter viscosity in relation to the development of the final cake structure, it is not known whether the more rapid increase in viscosity in the chlorinated sample is responsible for the improvement in the final cake structure.

Table 2. Viscosity (Poise) of batters made with flour chlorinated at two levels.

Temperature C°	Level of Chlorine (g/kg)	Viscosity (poise) after: ^a		
		5 min	10 min	15 min
76	0	15	20	23
	0.93	15	24	35
80	0	17	21	32
	0.93	20	36	49
85	0	18	65	124
	0.93	23	81	160
90	0	32	222	445
	0.93	35	267	532

^aCoefficient of variation is 10%.

Baking Properties - Temperature Profiles

Temperature profiles were recorded continuously at the positions previously described. Varying levels of chlorine did not create temperature variations greater than +4°C at any of the four thermocouple positions. Temperature gradients were established positionally within all the cakes irrespectively of chlorine treatment. The highest temperature was recorded by the edge thermocouple (6.9 cm from the center); the lowest temperature, generally at 2.5 cm from the center. This may be the result of the patterns of batter flow as described by Trimbo et al. (1966).

Figure 4 shows the center temperatures recorded across five levels of chlorine treatment. These center temperature curves show either three or four distinct sections: (1) an initial period of rapid temperature increase until the 8th min of baking corresponding to 83-86°C; (2) a period of less rapid temperature rise to the 17th min or to temperatures of 99-101°C; (3) a short period of intermediate temperature rise; and (4) a period of steady-state temperature after the 21st min with the temperatures approximately 111°C. This final period was not observed in all of the cakes, and its presence was not related to the level of chlorine treatment. Thus, there were no differences in the patterns of heat penetration that could be related to the level of chlorine treatment.

Water Loss Rates

The water loss rates versus time curves are shown in Fig. 5. The values for the rate of water loss as a function of baking time are the averages of three replications. The curves for all cakes show four distinct regions. During the first 4-5 min of baking, the systems begin to heat up, and water loss appears to be simply a function of temperature increase as if it is an isotropic system. At the end of this period, the temperature has

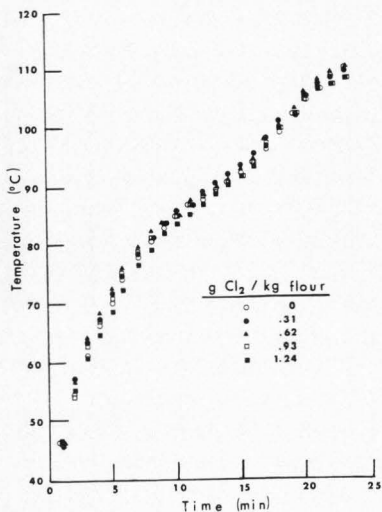


Fig. 4 Center temperature as a function of time during baking of batters containing 0 to 1.24 g Cl_2/kg of flour.

reached approximately 71°C . Changes then begin to take place within the batter that result in increased rates of water loss during the second period, which continues for 5–6 min. There were no observable differences throughout the first two sections of the curve across the level of treatments. The third section of the curve is characterized by a constant or decreased level of water loss. All the cakes entered this period at approximately 11 min into the baking time when the water loss rate is between 0.52 and 0.57 g/min. At levels below 0.93 g/kg, the water loss rate increased gradually during this period. It was not until the commercial level, 0.93 g/kg, that the water loss rate remained constant at 0.53 g/min for a period of 5 min. During this period,

the curve of the 1.24 g/kg sample in contrast to the other levels actually showed a decrease in water loss rate. The final period is characterized by an accelerated rate of water loss at all levels of chlorine. Thus, we can see that the effect of the level of chlorine in the water loss characteristics of the cakes during the baking process is more pronounced than the effect on the temperature gradient profiles.

Characteristics of the final cake

Table 1 summarizes the measurements made on the final cake as well as the pH and specific gravity of the batter. The areas of the cross-sectional tracings recorded as an index to cake volume in Table 1 suggest that increasing levels of chlorine treatment improve cake volume to a maximum corresponding to the 0.93 g/kg (commercial level use). The volume decreased at the 1.24 g/kg level, suggesting that the treatment is no longer optimal. The cake shrinkage indicators (diameter) reported in Table 1 show greater shrinkage at the lower levels of chlorine treatment. The minimum shrinkage was associated, again, with the 0.93 g/kg cake. Above this level, shrinkage of the cake again increased. The contour index reflects final shape of the cake. At the first two levels of treatment, the index reflects a sunken appearance. The surface of the 0.62 g/kg cake is relatively flat. At the commercial level of treatment, the cake is well-rounded. Above the commercial level, the contour index is less rounded. Based on the evaluation of these characteristics of the final cakes, the commercial level of treatment provided optimal results.

SEM of the cake crumb

SEM data were collected to determine differences in the cake crumb by evaluating swelling or matrix development in the final cake structure. The same positional effects reported by Gordon et al. (1979) were observed in all the cakes regardless of the level of chlorine treatment. Since these differences were not directly a function of the chlorine, attention was focused on the effect of the chlorine treatment on the structural development at a given position. The micrographs in Figure 6 show samples taken from the center bottom position across the five levels of chlorine treatment. Subjective evaluation of these micrographs is based on differences observed in the starch granules and in the matrix development. It is difficult to quantify these differences. For example, it is impossible to measure the degree of

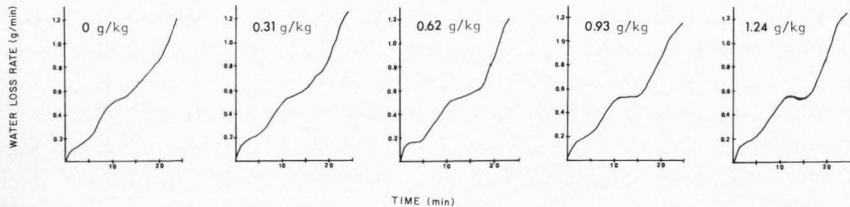


Fig. 5 Water loss rates as a function of time for batters containing 0 to 1.24 g Cl_2/kg flour.

granule swelling during gelatinization because of the limited number of granules viewed and the natural size distribution of the granules. As the matrix becomes more developed, the individual granules become less distinguishable. For this reason, less attention will be given to the size of the granules than to the more obvious structural differences. In the untreated sample (Figure 6a), the extragranular material forms clumps on the surface and between the starch granules. The granules themselves are intact, but have an amorphous appearance. At the 0.31 g/kg level of treatment (Figure 6b), there is evidence of the matrix being more fully developed. This material is thought to be made up of a combination of solubilized starch-lipid-protein. This lipid-protein-solubilized starch material begins to form a more continuous network between the granules. The next increment of treatment 0.62 g/kg (Figure 6c) shows further development of the matrix. The granules are still intact but appear less distinct as they become more embedded within the matrix. The matrix material also seems to have a cementing effect. The increased association between granules results in a greater buildup of the structural units reflected in the more three-dimensional appearance of the structure compared to the two lower levels. At the commercial level of the treatment (Figure 6d), the starch granules become less distinct as the matrix becomes more extensive. The formation of the larger building blocks due to the cementing effect of the extragranular material is still evident. The 1.24 g/kg level (Figure 6e) shows yet more extensive covering, making it impossible to identify the embedded granular structures. From this series, a most obvious structural difference at the center bottom position is the increased development of the lipid-protein-solubilized starch matrix with increasing chlorine treatment. This then acts to cement the components into larger building block units which will ultimately influence the final cake structure. The micrograph in Figure 7 shows two SEM photographs taken at the middle position of the untreated and commercial level treatments. The information gained by the higher magnification supports the findings in the lower magnification (Fig. 6). In the untreated sample, the extragranular material appears in clumps among the granules. Structure of the cake must then rely more on granule-to-granule contact. At the commercial level the extragranular material now forms a more continuous layer along the edge of the granule. The granules are more separated but seem embedded in a more solid matrix; therefore, they do not appear to function as individual units but as components of the larger building block units.

Discussion

It appears that the chlorination of soft wheat cake flours may have several effects upon the functionality of these flours when incorporated into a batter system for cake formulation. It appears that particle size needs to be increased to an optimum size in order to give a better structure in the cake; however, it is not yet

known if the increased size observed in the dry particles is responsible for the increased association of the components in the final cake. From the preliminary data on flour component chlorine uptake, there is indication that chlorine:sulfur ratio for the protein component is more responsive to increasing levels of chlorination than is the chlorine:potassium ratio in the starch component. If there is indeed preferential uptake of chlorine by the protein component in the chlorinated cake flour, this might explain, in part, the increase in the viscosity and coincides with extensive matrix development when either flour-water slurries or total batter components are heated. This would then account, in part, for the greater viscosity that one sees with the Brabender Amylograph measurements and with the Haake Rotoviscometer. It does not exclude, however, the possibility that chlorine could affect the starch itself by influencing the ability of the amylose to be leached out of the starch granule and to complex with the protein component of the flour. Therefore, the earlier increase in viscosity with chlorination may aid in better structural development during the baking process, resulting in maximum volume and contour, and in less shrinkage of the baked cakes. These indices of good cake structure (maximum contour and minimized shrinkage) are reversed when we exceed the commercial level of chlorine treatment of the flour. At this time, it is not obvious why this reversal should occur. Temperature profiles during the baking process do not seem to be affected by level of chlorination. In other words, at any moment in time during baking, temperatures in the different positions are similar whether the batters are made from treated or untreated flour. Some dramatic differences, however, are seen in the water loss characteristics during the baking process. These might be due to purely physical differences such as the increasing viscosity, which may impair the water movement out of the cake or to differences in crumb pore structure. The water loss characteristics may also reflect differences in starch gelatinization and/or protein denaturation patterns or complexing behavior that result from the response of starch lipid or protein fractions to chlorination and subsequent heating in the batter system. These changes could affect the water and energy requirements needed for these transitions and interactions to take place. However, at levels exceeding the commercial level of chlorine treatment, the rate of water loss still shows a local maximum in which the rate of water loss decreases for a short time period within the temperature range of the starch phase transitions. Why this happens is not well-defined or understood at this time. It might be that the overdevelopment of the matrix results in more of a barrier to the movement of water out of the system. Further experiments will need to be done in order to better evaluate the meaning of the water movement inhibition during the period of starch phase transitions, because prior to and after that period the rates of water loss appear similar.

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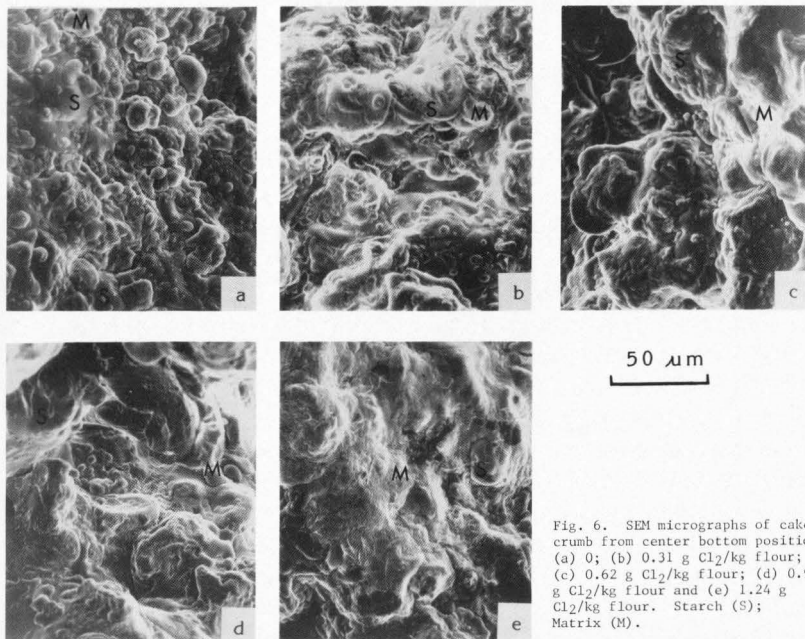


Fig. 6. SEM micrographs of cake crumb from center bottom position: (a) 0; (b) 0.31 g Cl₂/kg flour; (c) 0.62 g Cl₂/kg flour; (d) 0.93 g Cl₂/kg flour and (e) 1.24 g Cl₂/kg flour. Starch (S); Matrix (M).

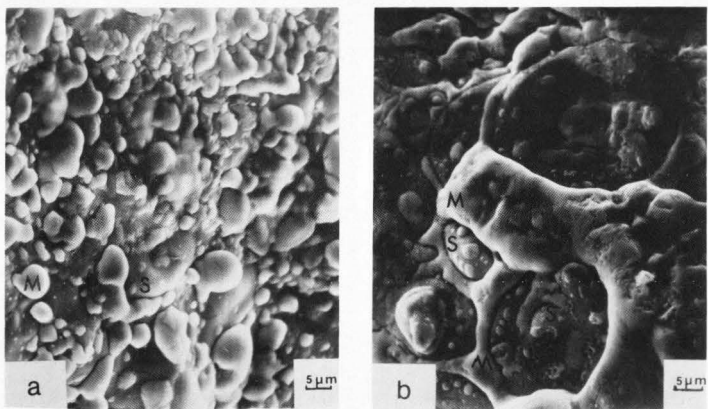


Fig. 7. SEM micrographs of middle position: (a) 0 g Cl₂/kg flour; (b) 0.93 g Cl₂/kg flour. Starch (S); Matrix (M).

Acknowledgements

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Discussion with Reviewers

R. Moss: Why do the readings of temperature and water loss rates stop at 23 minutes although the baking time is 25 minutes?

Authors: The design of the oven is such that there is a two minute lag-time during which the water vapor clears the oven and the wet-bulb, dry-bulb temperatures are recorded. The time scale is corrected for this, but the data for the last two minutes of baking cannot be recorded. For details, see Godsolve et al. (1977).

W.J. Wolf: Have you attempted to use TEM in these studies?

Authors: Yes, preliminary studies from unpublished freeze fracture data show that unheated starch granule surfaces from wheat flour that has been treated with 1.24 g/kg are smoother than those of starch granule surfaces from flour that has not been chlorine-treated. Generally speaking, the starch granule cross-sections from untreated and unheated granules appear to show a ridge around the circumference that support the surface granule observation. Also, after heating up to 102°C, the overall degree of swelling is not noticeably different between 0 and 1.24 g/kg chlorine treatment, although some lamellar-type layering seems to be present in the untreated starch granules as they swell. Further work is being done to verify these observations.