

APPLICATION OF THE CHOPIN ALVEOGRAPH
TO THE STUDY OF STRUCTURAL RELAXATION IN DOUGH

by

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ABSTRACT

A method, analogous to that used for extensograph, was developed for the study of structural relaxation in bromated and unbromated doughs using the physical dough testing instrument, the Chopin alveograph.

A mathematical analysis of the alveogram, including a consideration of the geometry of alveograph bubbles was carried out to establish a fundamental measurement by which groups of alveograms may be analysed for the construction of relaxation curves. This analysis revealed that the pressure within a dough bubble is directly related to the thickness of the membrane of the dough bubble. The pressure measurement, referred to a constant sample deformation i.e. a constant membrane thickness, was found to be a fundamental measurement suitable for the construction of relaxation curves obtained from alveograms under a wide variety of conditions.

Several other standard measurements including the alveogram maximum, square root of the volume, area under the alveogram curve, extensibility and the work function were examined under a wide variety of conditions. Only one of these measurements, the extensibility, could be applied to the study of structural relaxation in doughs.

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INTRODUCTION

The physical properties of bread dough are becoming increasingly important as a criterion of flour quality. The elastoviscous properties of dough are utilized to assess flour absorption, baking strength and optimum flour improvement. Equally significant is the fact that information about the molecular structure of dough may be obtained by a study of its physical properties.

A variety of mechanical dough testing machines have been designed primarily to evaluate flour quality. One of these instruments, the extensograph, measures the resultant of numerous dough properties by recording a load-extension curve of a sample of dough which is stretched to the breaking point. Early investigators compared the length, maximum height and area of the load-extension curves to determine the baking strength of different flours. However little basic knowledge could be obtained from these curves because the mechanical properties measured by the apparatus were not defined in meaningful fundamental units.

Dempster, Hlynka and Winkler (7) have devised a more basic method of using the extensograph to study the physical changes taking place in doughs. This method has introduced the following new concepts into dough chemistry.

- (a) A constant sample deformation was introduced as a common basis of comparison for all samples.
- (b) A reaction time was permitted for the reaction between dough and other reagents used.
- (c) The dough was given structural activation in order to make it possible to measure the effect of the reagent.
- (d) A rest period was provided during which the dough was

allowed to "relax".

These terms will be more fully defined in the text of the thesis and it is sufficient at this point to state that the change in physical properties of bromated and unbromated doughs were summarized in the form of a structural relaxation curve which was then described by its specific constants.

The object of this research is to investigate the possibility of using another physical dough testing machine, the Chopin alveograph, for the study of the structural relaxation process in bromated and unbromated doughs. This apparatus measures dough properties by recording the pressure required for the extension of a small sample of dough blown into the shape of a bubble. There are two fundamental differences between the alveograph and the extensograph. These are (a) the extensograph stretches the dough in one direction whereas the alveograph stretches the dough in two directions (b) the extensograph stretches the dough at a constant rate which is independent of the resistance of the dough to stretching, whereas the rate at which the alveograph bubble expands and stretches the dough must be affected by the resistance of the dough to stretching. The alveograph, unlike most physical dough testing machines, is imitative of the actual breadmaking process. The two dimensional deformation of dough to form a surface is comparable to the fermentation in bread dough in which the liberated carbon dioxide causes small bubbles to be blown through the dough structure.

The standard measurements made on the alveograph by early investigators including Chopin (6), Scott Blair (17) and Potel (17) and others represented a complex group of variables and are not significant in obtaining any fundamental information in relation to molecular dough

structure. Because of the differences between the alveograph and the extensograph it is reasonable to expect that the alveograph will bring out certain aspects in the study of the structural relaxation in dough that cannot be observed with a similar study on the extensograph.

A major part of this thesis will be devoted to a study of the factors involved in applying the method of Dempster and co-workers to the Chopin alveograph. A comprehensive examination will be made of the conditions necessary for a constant sample deformation, reaction time, rest period and structural activation in relation to the problems they present as applied to the alveograph. The remainder of this thesis will present a variety of illustrative results of structural relaxation studies in doughs based on the alveograph.

REVIEW OF LITERATURE

Fermentation in bread can be described as the transformation of a dough from a homogenous mass to a light heterogeneous sponge-like material. In this process the dough is transformed into a cellular or porous structure which is brought about by the yeast through the liberation of carbon dioxide gas into the cells. The carbon dioxide gas exerts a pressure which inflates the dough by blowing many small bubbles, not of uniform size or distribution, throughout its structure.

Realizing the need for a machine to study this phenomenon, Chopin (6) invented an apparatus called the alveograph. This apparatus provides a mechanical test on dough which corresponds, in a general way, to the deformation which it would undergo in forming a loaf.

An operational description of the alveograph is provided by Chopin (6). A small dough sample is turned down into a thin membrane on the alveograph. Air is forced under the sample causing the membrane to be inflated into the shape of a bubble. The pressure changes taking place during the expansion until the bubble ruptures are recorded on a kymograph. A detailed description of the pressure-time curve (alveogram) obtained, along with a full discussion of the apparatus, will be given under the section entitled "Materials and Methods".

A summary of the procedure followed by Chopin for preparing test samples is related here. Three hundred and fifty gm. of flour is placed in a mixer which turns at the rate of 50 revolutions per minute at a

temperature of 20°C. The dough is mixed for 20 minutes, placed in a cylindrical mold and rested for 20 minutes at 20°C. It is divided into eight separate samples and tested immediately.

In order to facilitate the dough mixing for test samples on the alveograph, Chopin invented a dough mixing machine. This machine was designed in such a way such that the force exerted in mixing the dough is registered by a recording device. This force was assumed to be a function of the relative plasticity of the dough and changes progressively during long mixing periods. After the dough is mixed sufficiently it is extruded through a narrow slot 6 mm. wide. The extruded dough is then ready to be cut into an appropriate size used for the test samples.

Chopin describes a method for calculating the specific energy of deformation W. He assumed that the total amount of work done from the start of the bubble to the final rupture could be represented by:

$$T = \int_0^V PdV$$

where dV is an infinitesimal increase in volume

P = pressure of air inside the bubble
at that instant

V = final volume

T = work done

Chopin assumed that the specific energy of deformation along with the coefficient of extension were sufficient to characterize the viscoelastic properties of the flour. Chopin carried out an investigation with different flours in which he determined the coefficient of extension and the specific energy of deformation. He found that the specific energy of deformation varied considerably depending on the nature of the wheat.

Chopin proposed that a simple relation existed between the ability which a flour possesses of being stretched into a thin membrane and the specific loaf volume when this flour is baked into bread.

During the course of his investigation Chopin determined the "tenacity" and "coefficient of extension" of the dough using the alveograph. The maximum pressure (in mm. H₂O) was assumed to be proportional to the tenacity of the dough. The surface area of the membrane at the time of rupture was taken as a measure of the coefficient of extension of the dough represented by E. The volume of air was represented by V. A number of experiments were carried out to measure the tenacity of the dough (P in mm. H₂O) and E, the coefficient of extension which was assumed to be proportional to V. A simple relation was found between the loaf volume and the coefficient of extension E.

$$V - V_0 = K\sqrt{E}$$

where V_0 = initial volume of dough

V = final loaf volume

K = a constant

This relation is a very general one as shown by the graphs plotted. These graphs did not show a very good relationship. The equation implies that the difference between the specific volume of the bread which may be obtained with a flour, and the initial specific volume of this flour dough is proportional to the square root of the coefficient of extension of this dough.

Chopin found that the amount of water absorbed by a dough was proportional to the tenacity P, such that a small value of P indicates that a flour has a low absorption, whereas a large value of P indicates a flour with a high absorption.

Bailey and Le Vesconte (3) extended Chopin's work by carrying out the following lines of research with the alveograph (a) the effect of prolonged mechanical treatment in mixing the dough (b) increasing the proportion of water in the dough (c) adding starch to the flour (d) varying the hydrogen ion concentration of the dough (e) treating the flour with chlorine (f) adding flour improvers (g) fermenting with yeast (h) correlating extensibility readings with the results of baking tests of several flour samples.

The dough samples were prepared in the following manner: a 350 gm. dough was mixed for eight minutes using a salt concentration of 1.4 gm. salt per 100 gm. of flour. The dough was then rolled to a thickness of 18 mm. and let stand for 25 minutes. Test pieces were then taken from this dough and tested in the alveograph kept at a temperature of 25°C. It was found by these investigations that the extensibility of the dough was decreased appreciably by increasing the time of mixing from eight minutes to sixteen minutes and continued to decrease with a longer period of mixing.

The effect of extensibility by varying the absorption from 59% to 67% was investigated. Bailey and Le Vesconte found that a dough of 67% absorption was too soft to be handled easily and results obtained showed a large variation. The extensibility was found to increase up to an absorption of 64% at which point further increase in absorption gradually reduced the readings.

Starch was found to have a pronounced effect on the extensibility of the dough. The extensibility of the dough was found to diminish with increasing starch concentration.

Bailey and Le Vesconte measured the effect of pH on dough extensibility. The dough was found to be most extensible at a pH of 6.1 and the extensibility was lowered for a pH below 5 or above pH 7.

The effect of certain chemical reagents on the extensibility was studied and it was found that calcium acid phosphate improved the extensibility in every case, phosphoric acid was noticeably detrimental to extensibility and calcium sulfate, calcium peroxide and magnesium sulfate lowered the extensibility.

Bailey and Le Vesconte assumed that fermentation would decrease extensibility due to the fact that the hydrogen ion concentration increased during the fermentation process. After carrying out an investigation using different fermentation periods the assumption appeared to be correct since the extensibility decreased with increased fermentation time.

Scott Blair and Potel (17) describe the physical significance of certain properties measured by the Chopin alveograph. These investigators gave their views as to what occurs when a bubble is blown from a small patty of dough. The air pressure required to blow the bubble is obtained by the displacement of air by a rising water column and this pressure is applied at a regular but decreasing rate. Scott Blair and Potel state that although the pressure appears to pass through a maximum there is no reason to believe that the stress per unit area does not continue to rise up to the final rupture. It has been experimentally shown that

the viscosity of flour falls with rising stress (structural viscosity) but rises with increasing deformation (18). Scott Blair states that the variation of viscosity during the production of the bubble is complex but at the point of final rupture the viscosity may be defined as the momentary shearing stress divided by the rate of change of non-recoverable deformation. Accordingly, the value of pressure P could be correlated with the viscosity of the dough. Since the type of absorption of a dough determines the viscosity of a dough, it can be assumed that the value of P , being related to viscosity, is a measure of the water-absorbing capacity of the flour.

These investigators give an account of the significance of the square root of the volume (G) of the bubble. Scott Blair and Potel divide the total deformation into two parts (a) recoverable deformation (σ_E) and (b) non-recoverable deformation (σ_n). σ_E is defined as the ratio of the shearing stress S to the shear modulus n .

where n = modulus

η = viscosity

Therefore the lower the modulus the larger the value of σ_E for a given stress. Also the higher the viscosity, the larger the stress under a fixed rate of deformation. Consequently, the higher the viscosity, the greater the proportion of elastic to total deformation. Therefore a high value of η and a low n are the most significant factors in producing a high value of G . Halton and Scott Blair (12)

pointed out that some measure of extensibility is obtained from the

ratio $\frac{\eta_h}{\eta_l}$

where η_h = viscosity at high stress

η_l = viscosity at low stress (normal stress)

Relying on the above assumptions Scott Blair and Potel wrote a generalized equation for the value of G namely

$$G = (f) \frac{\eta_l^a}{\eta_b} \cdot \frac{\eta_h^c}{\eta_l^d} \quad (1)$$

where a, b, c, and d are unknown powers.

Scott Blair and Potel tested 35 doughs by the standard Chopin technique and obtained the following P, G, η_h , η_l and η . P and G were determined in the usual way. η_h was derived by measuring the rate of flow of a dough under a load of 80 to 85 kilograms per cm.² through a narrow brass tube of 5 mm. diameter and 5 cm. length. η_l and η were determined by the method used by Halton and Scott Blair (19, 12) at a shearing stress of 1200 dynes per cm.².

The validity of equation (1) was tested by assuming that a = d and c = 1 giving the following:

$$G = (f) \frac{\eta_h}{\eta_b} \quad (2)$$

This equation was found to be valid for a value of b = 2.0. Two graphs were plotted to show the correlation between P vs. η_l and G vs. $\frac{\eta_h}{\eta_b}$.

Aitken, Fisher and Anderson (1) made a study of the type of alveograms obtained from various types of wheat flours. They experimented mainly with two series of wheat, namely a series that varied widely in both protein content and baking strength and a series of wheat that was essentially the same in these respects. Tests were also made with the extensograph and farinograph but these results are not included in this review.

The tests were carried out using a fixed absorption (48%). Dough containing 1 gm. salt per 100 gm. flour (13.5% moisture basis) was mixed for 6 minutes in the Petrin extractor. The dough was extruded and cut into 5 discs of uniform size. The discs were rested in a constant temperature (25°C.) oven for 20 minutes and then tested on the alveograph. Three measurements were taken from each alveogram, maximum height (cm.), maximum length (cm.) which measures extensibility of the dough, and the area under the curve. The total work W in ergs was computed from the relation $\frac{K \times C \times S}{L}$ where K is a constant (manometer correction coefficient), C a volume associated with the volume of liquid in the gasometer, S the area under the curve, L the length of the curve in cm. The error for the average of five replicate curves was found to be ± 0.4 cm. for an average length of 11 cm. and ± 0.02 cm. for an average length of 8.5 cm.

After carrying out numerous tests with the alveograph it was found that as the protein content increases there is a regular increase in length of the alveogram from 7.2 to 12.4 cm. and a regular decrease in height from 10.0 to 7.9 cm. The work W , was found to increase from 325 to 428 units. These investigators stated that the extensibility of the alveogram is closely related to protein content.

Bennett and Coppock (5) describe a new technique by which the Chopin alveograph may be utilized for detecting the effect of improvers in flour. Working on the assumption that the character of the dough may be changed by manipulation of the dough they developed a new type of moulding technique in order to bring out the effect of improvers on flour dough using the alveograph.

This technique may be summarized in the following manner: the flour is made into a dough using 1 gm. salt per 100 gm. flour. The dough is mixed for seven minutes. The dough is placed in a constant temperature oven at 26.7°C. for 3 hours. Four 20 gm. balls are weighed and each ball is moulded by giving it 40 revolutions in 12 sec. on a special moulding unit which consists of a smooth block of wood, a small nail, a biscuit cutter and a glass funnel. The moulded ball is flattened into a disc to a thickness of 0.5 cm. The discs are placed on a shelf in a constant temperature cabinet for 20 minutes before testing.

Several improvers were tested using the new moulding technique and times indicated. These improvers include potassium bromate, ascorbic acid, nitrogen trichloride (agene), chlorine dioxide (Dyox). Potassium bromate samples tested by the standard method in concentration of 0.002%, 0.004%, 0.006% showed very little differentiation between the curves. However, doughs treated with 0.001%, 0.006%, 0.002% bromate showed large differences in heights of alveogram curves when subjected to the revised moulding technique.

The effect of Agene and Dyox was noted after testing doughs treated with these chemicals by both methods. It was found that under the standard method doughs treated with Agene and Dyox tend to increase the height and shorten the length of the alveograms. Doughs treated by the moulding technique gave the same type of curve as the standard method only to a more pronounced degree. Ascorbic acid shows little effect on the flour with the standard procedure. However, the curves are progressively higher with increase in concentration of ascorbic acid when the doughs were subjected to the moulding technique.

Bennett and Coppock investigated the relation between time and moulding in curves obtained on bromated dough. It was found that the bromate effect is noticeable only if a resting period before moulding is allowed. Consequently, doughs which do not have a resting period and are not subjected to moulding do not show the bromate effect. These curves showed that the longer the resting period before moulding the greater is the bromate effect.

Baking tests were also performed to show the effect of potassium bromate on bread. It was found that the greater the concentration of bromate the greater the size of the loaf.

Historical accounts of the alveograph and its operation are given by A. J. Amos and D. W. Kent-Jones (15), G. W. Scott Blair (11) and C. H. Bailey (4). The accounts given by the above named do not add any new information to the work that has already been done in this field and therefore only mention is made of them. A review of the history of the alveograph is also given by a bulletin entitled "A Review of Methods for Determining the Quality of Wheat and Flour for Breadmaking" edited by the Agricultural Experiment Station, Kansas State College(16).

A series of papers have been published concerning another empirically designed dough testing instrument called the Brabender extensograph. The work done by Dempster, Hlynka, Anderson and Winkler (7, 8) on this instrument is, as will be seen later, pertinent to the investigation to be carried out with the alveograph.

The extensograph measures the resultant of a variety of physical dough properties by recording a load-extension curve or extensogram as the sample of dough is stretched at a constant rate. The time dependent structural changes which take place in dough are demonstrated by the extensograms. For example, a short high extensogram is typical of a freshly mixed dough stretched soon after mixing. On the other hand, a long low extensogram is typical of a "rested" dough, i.e., a dough which has been given a long period of time between mixing and testing.

Dempster et al. (7) have devised a method by which the time dependent changes in bromated and unbromated doughs can be observed. These changes are examined by plotting the load, recorded on extensograms for a constant sample deformation, vs. rest period. The curve obtained is a graphic representation of the relaxation process which takes place in doughs and is therefore called a structural relaxation curve.

The most significant factors which are necessary to obtain the relaxation curves are : (a) a reaction time, i.e., the time between mixing and shaping the doughs, (b) structural activation in shaping and rolling the dough on the extensograph, (c) a rest period, i.e., the time between shaping and stretching the dough on the extensograph.

The method developed by Dempster et al. for the construction of relaxation curves will serve as a basis from which a method will be developed for the structural relaxation study of bromated and unbromated doughs using the alveograph.

MATERIALS AND METHODS

The materials used in this study consisted of one commercially milled flour from a blend of Canadian spring wheats grades 1 and 2. The absorption of this flour was 61.2%. This absorption, on a 14% moisture basis, was determined by the farinograph using a dough consistency of 540 farinograph units. The ash content was 0.44%. The protein content was 13.0% as determined by the Kjeldahl method (16) recorded as nitrogen x 5.7 and corrected to a 14% moisture basis.

The principle underlying the use of the alveograph involves the inflation of a dough membrane by air pressure into a spherical bubble and the recording of the pressure changes taking place during the inflation of this bubble.

Figure 1 shows a photograph of the Chopin alveograph. For purposes of illustration this apparatus can be divided essentially into three major parts:

(a) A dough press in which a patty of dough is pressed to a thickness of 0.25 cm. This press acts as a "bubble platform" or in other words, a platform from which the dough membrane is inflated into a bubble. The photograph shows a bubble emerging from the center of the dough press. A squeeze bulb on the left hand side of the press is used to raise the patty off the surface of the base plate before expansion begins.

(b) A glass burette \approx 1000 cc. capacity, from which air is displaced by water to cause the expansion of the dough membrane. This part of the apparatus appears directly behind the dough press

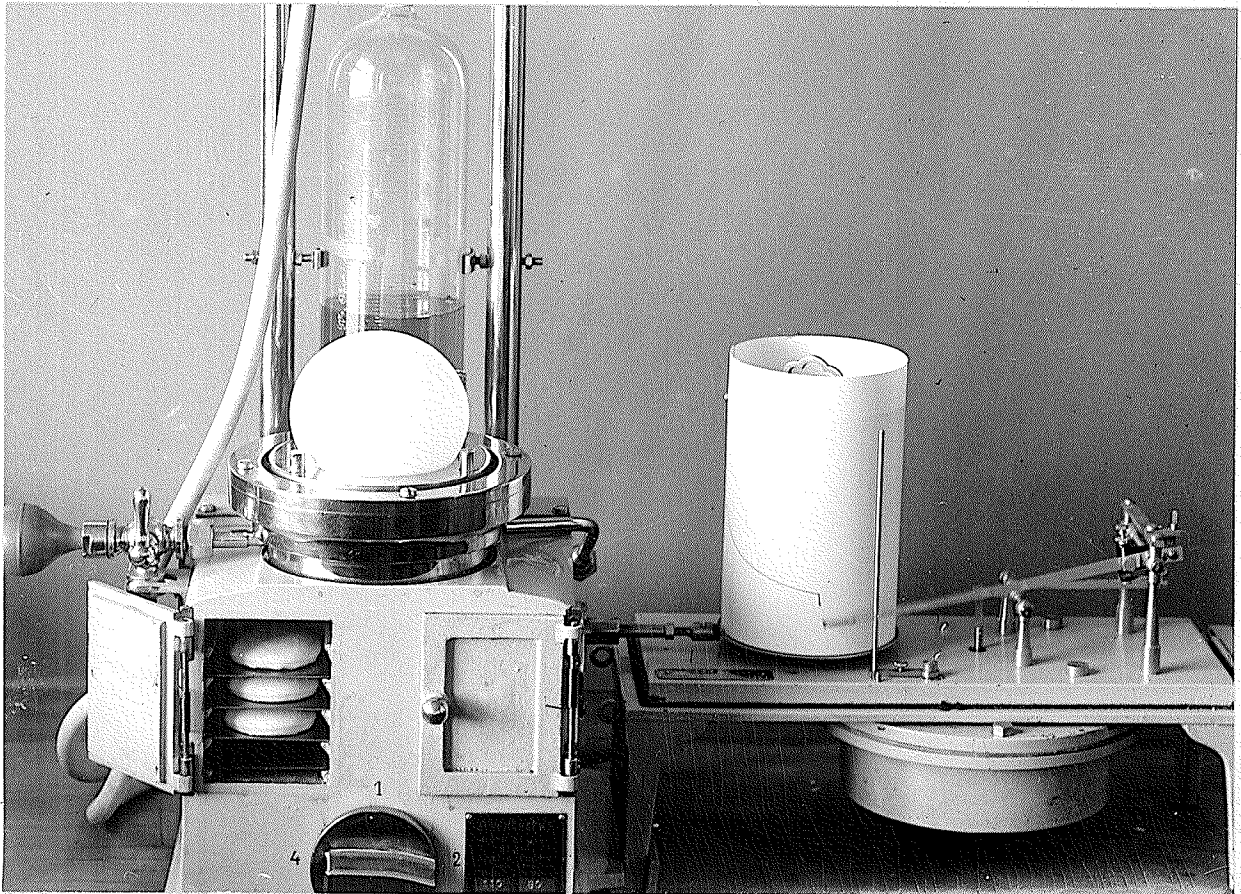


Fig. 1

The Chopin Alveograph: a test dough is being expanded into a bubble and the alveogram recorded.

in the photograph. Two metal pillars hold the burette in position and support, on a platform, a water bottle directly above the burette. Water flows from the water bottle into the bottom of the burette through a rubber tube forcing the air from the burette. This burette is calibrated in units of square root of volume.

(c) The pressure recording unit, at the right in the photograph, draws a pressure extension curve of the bubble blowing process.

In operation, a patty of dough is placed on the oiled base plate of the dough press. A larger circular clamp is then placed over the dough patty and screwed down until the dough attains a thickness of 0.25 cm. The upper surface of this clamp consists of a removable brass disc held in place by an annular screw. When the screw and disc are removed after the clamp has been screwed down, the dough patty is ready for the expansion into a bubble. The handle (shown below the cabinet door in the photograph) controls the water flow into the burette and the air flow to the dough bubble. Normally the handle is in position (1). When in this position the water in the burette is adjusted to zero reading by varying the height of the water bottle. This lever is then turned to position (2). The water bottle is placed on the bracket above the burette. The mechanism of the revolving drum (right hand side of diagram) is engaged. When the handle is turned to position (3)

(opposite position (1)) the water rises in the burette causing air to be forced under the membrane which is inflated into a bubble. The pressure extension curve is recorded on the drum which is set into motion by releasing a brake the instant the handle is turned to position (3). When the membrane ruptures, the process is ended and the handle is turned quickly to position (4). The volume of the water in the burette, which is taken as equal to the volume of the bubble, is recorded.

Figure 2 shows a typical pressure-extension curve or alveogram obtained from a test made on the alveograph. Certain definite measurements connected with this curve have been reported in the literature. These measurements have been used as indices of flour quality.

In Fig. 2, BC gives the length of the alveogram which is taken as a measure of the extensibility of the dough. The maximum height of the curve AB, is taken as the initial tensile strength of the dough. The height CD, is taken as the tensile strength at the time of rupture. The work function, derived from the expansion of the membrane, is determined from a calculation of the area under the curve. The work function is also known as the specific energy of deformation of the dough and the square root of the volume as the coefficient of extension of the dough.

Now that the operation of the alveograph has been described a review of previous methods of using this apparatus will be given. This review of methods will serve as a starting point from which a method that can be applied to the bromate study will be developed.

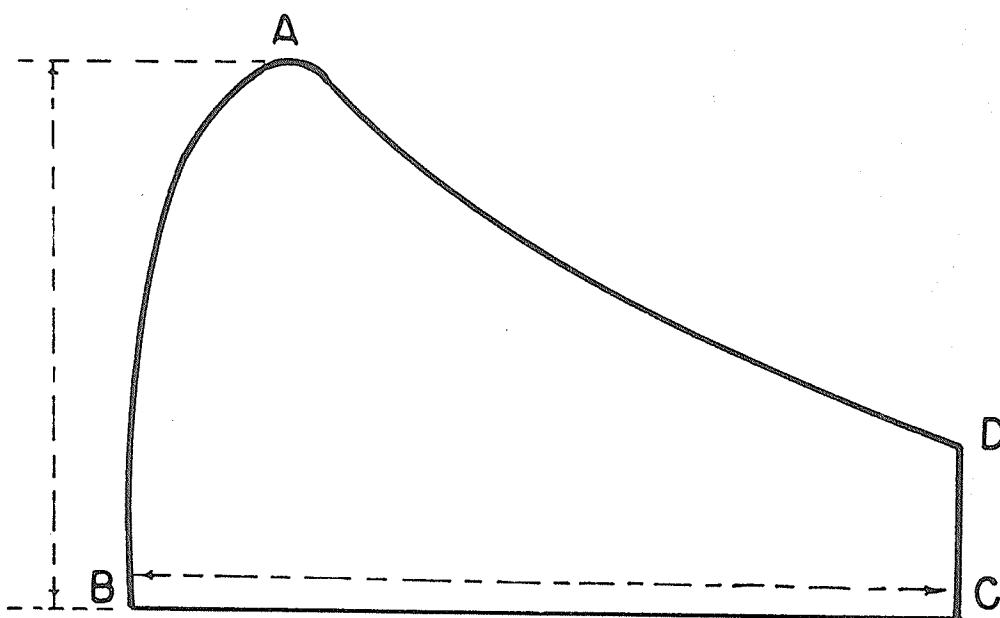


Fig. 2

Some standard measurements
made on a typical alveogram.

The first method adopted for use on this instrument was given by Chopin (6). Amos (2) has described a method of using the alveograph which was, for all intents and purposes, a summary of that used by Chopin and which has become known as the standard method. A description of this method is now given: a dough is mixed from 250 gm. of flour using a salt concentration of 1 gm. salt per 100 gm. flour. A strip of dough is extruded through the slit of the Petrin extractor, the dough is cut into sections which are rolled into standard thickness (method of rolling not mentioned) and cut into discs. Four discs are made from each dough and placed in a constant temperature cabinet at 25°C. for 20 minutes. The discs are tested on the alveograph in the manner described previously.

Bennett and Coppock (5) revised this standard method in such a way that the effect of bread improvers such as potassium bromate could be studied qualitatively by using the Chopin alveograph. Previous to their work the effect of bread improvers could not be shown on the alveograph curves using the standard method. Curves obtained from bromated doughs showed little or no differentiation from those obtained from unbromated doughs. Consequently, using the standard method, a study of the bromate effect could not be made.

The moulded method consisted of making a dough using 1% salt concentration (flour weight) and a mixing time (using the Petrin extractor) of seven minutes. The dough is removed from the mixer and placed in a constant temperature cabinet kept at 26.7°C. for a 3 hour period. At the end of this period four 20 gm. dough balls

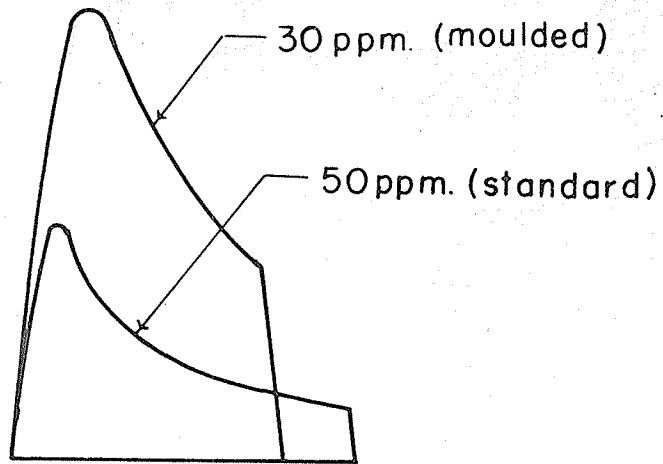


Fig. 3

Alveograms showing the effect of potassium bromate on doughs treated by the "moulded" and standard methods.

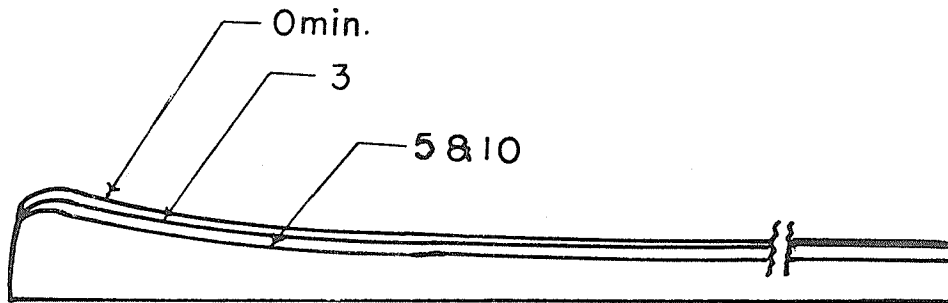


Fig. 4

A set of alveograms given rest periods of 0, 3, 5 and 10 min. showing structural relaxation after a 1 hr. reaction time.

are scaled off and weighed. Each dough ball is moulded by giving it 40 revolutions in 12 sec. on a specially constructed moulding unit. The dough ball is flattened between two oiled glass plates to a thickness of 0.5 cm. The patty thus formed is allowed a rest period of twenty minutes. Figure 3 shows the effect of the moulded technique compared with the standard technique. The upper curve was obtained from a dough containing 30 p.p.m. KBrO_3 and treated by the moulded method. The lower curve was obtained from a dough containing 50 p.p.m. KBrO_3 treated by the standard method.

The investigations with bromate made by Bennett and Coppock can best be understood from a study of the basic rheological concepts introduced by Dempster, Hlynka and Winkler (7). They have found, using the Brabender extensograph for the bromate effect in dough, a "reaction time" must be allowed. This factor may be defined as the time which elapses between mixing and moulding the dough. A reaction time is necessary so that the bromate will have an opportunity to react with the dough, since it has been shown that the bromate effect is latent. Furthermore, the dough must be mildly manipulated by rolling or rounding to show the bromate effect. This manipulation has been called structural activation by Dempster et al. After the dough has been activated it is allowed to "rest" a certain time before testing. This time is called a rest period and is defined as the time between moulding and testing the dough. Dough assumes an activated or higher energy state after moulding. The time dependent physical changes, which take place as the dough returns from a highly activated state to a more stable state of lower energy, are grouped under the term of structural relaxation of the dough.

The moulded method of Bennett and Coppock shows the bromate effect for a flour under one set of conditions, namely at a reaction time of three hours and a rest period of twenty minutes. To study the structural relaxation of dough due to the bromate effect it is necessary to test samples of dough at a variety of rest periods and reaction times. Consequently, a method which will provide for a variety of reaction times and rest periods is needed for the study of the bromate effect on the alveograph.

Dempster, Hlynka and Anderson (8) devised a method, which includes the above requirements, to study the bromate effect using the Brabender extensograph. A method analogous to this has been adapted for use with the alveograph. The method provides for essentially three main factors (a) a reaction time (b) structural activation of dough and (c) a rest period. Certain modifications had to be made for these three factors to be utilized for the bromate study with the alveograph. These changes include (a) revised moulding method (b) modification of alveograph apparatus (c) a study of activation of dough.

No significant changes had to be made in reference to the reaction time given to any particular dough sample. The reaction times used in the extensograph method were adopted for use with the alveograph.

However, the apparatus used for activation of the dough samples had to be modified. The apparatus used by Bennett and Coppock consisted of a glass funnel under which the dough was impaled by a small nail. This funnel rotated on a wood surface around the circumference of a circular cookie cutter. Doughs given too long rest periods became

soft and sticky and could not be rounded uniformly because they would become attached to the side of the glass funnel in the moulding process. To obtain the greatest uniformity in moulding for dough under all conditions, the design for the rounding apparatus used on the Brabender extensograph was adopted.

A photograph of the moulding apparatus used is given in Fig. 5. The apparatus consists of a small metal box 5 cm. by 5 cm. open at both ends enclosed in a metal ring 8.7 cm. in diameter. The dough ball is tethered to a small nail at the center of a large metal ring (inside diameter 12.8 cm. and outside diameter 16.6 cm.). The metal box equipped with a 300 gm. lid is placed over the dough ball and the box rotates within the large metal ring. The plate on which the ring is placed, is kept at 30°C. by heat conduction through the metal from a heated water bath. All other parts of the moulding apparatus are temperature controlled at 30°C. in a constant temperature cabinet.

It was found that doughs given long rest periods (30-45 minutes) became very extensible, blowing very large bubbles when tested on the alveograph. The glass burette provided with the alveograph did not have a large enough capacity to measure the volume of these bubbles. For this reason a plastic water tight cylinder with a capacity of \approx 2000 cc. was substituted for the glass burette. This plastic cylinder is large enough to measure the volume of any size bubbles of practical interest that can be inflated on the alveograph. The original water reservoir (capacity \approx 1000 cc.) had to be replaced by a 2 quart jar of capacity \approx 2000 cc.

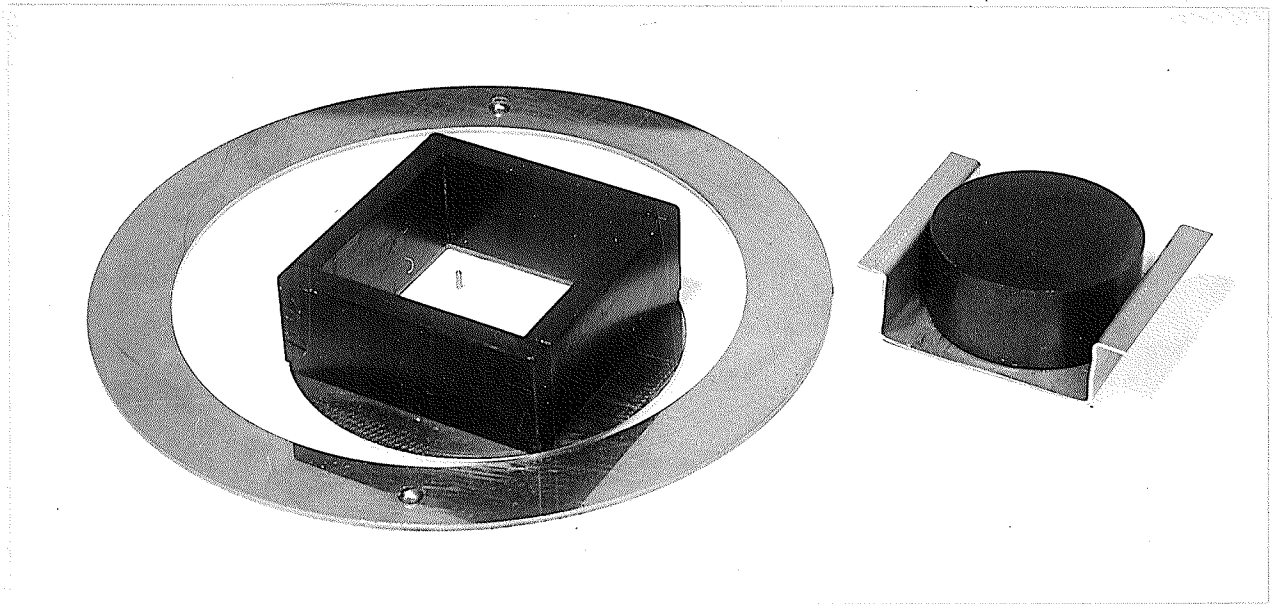


Fig. 5

A photograph of the moulding apparatus showing metal box, weighted lid and metal ring.

To develop a suitable method to study the bromate effect on dough with the alveograph it is necessary that no other form of activation take place other than the moulding process. Consequently, experiments were carried out to determine whether any activation was caused from turning down a patty of 0.5 cm. thickness to a thickness of 0.25 cm. in the dough press. This investigation was carried out in the following manner: 100 gm. of flour was mixed for 3 minutes in the GRL mixer (13). Twenty gm. were then scaled off, moulded and pressed into a patty 0.5 cm. thick. The patty was then given a rest period of one hour. During this period the structural activation due to the mixing and moulding process had time to be, for all practical purposes, completely dissipated. The same procedure was carried out for other dough samples giving them secondary rest periods of 0, 3, 5 and 10 min. in the dough press of the alveograph before testing.

Figure 4 shows a set of curves obtained for the dough samples given secondary rest periods. The low curves are typical of a very relaxed dough. This point is further emphasized by the fact that the 0, 3, 5 and 10 min. curves are very close together showing that the activation of the dough caused by moulding has been dissipated.

A secondary rest period of 3 min. in the dough press was adopted. This rest period was chosen because "0 minute" rest period gave a slightly higher curve which indicates a slight activation, and for longer rest periods of 5 and 10 min. the heights of the curves were very similar to that of the 3 min. rest period. Thus no appreciable activation was noted for secondary rest periods of 3 min. or longer.

The secondary rest period given to the dough is included in the initial rest period (the period between moulding and testing the sample).

The thickness of the dough patty was reduced from 0.5 cm. to 0.25 cm. to reduce further any activation effect in the dough when the patty was turned down in the press.

Having made the adjustments just mentioned the method for using the alveograph for the study of the bromate effect on dough may be described.

One hundred gm. of flour are doughed using 1% NaCl by weight. The dough ingredients (temperature conditioned at 30°C.) are mixed in a specially constructed GRL mixer (13) for exactly 3 minutes. The dough is removed from the mixer and placed in a constant temperature (30°C.) humidity cabinet for a reaction time of 0, 1, 2, 3 or 4 hr. Doughs are given a "0" hr. reaction time in which case they are activated and then tested immediately after the appropriate rest period on the alveograph. After the reaction time has elapsed one 20 gm. sample is scaled off the dough, weighed and moulded, giving the dough 30 revolutions in 12 sec. on a specially designed moulding unit. The dough is then flattened to a thickness of 0.25 cm. on an oiled metal plate for 10 seconds by a large metal plate. It must be remembered that the actual thickness of the dough patty is greater than 0.25 cm. due to the fact that although the dough may be initially pressed to a thickness of 0.25 cm. when the plate is removed, the dough "springs" back assuming a larger thickness. The patty is now placed in the

humidity cabinet where it is given a rest period of 5, 7, 10, 20, 30 or 45 min.

Two minutes are allowed between the end of the reaction time and the beginning of the moulding of the dough. The rest period begins right after the dough is flattened into a patty. The dough patty is removed from the cabinet at the end of the rest period, placed in the alveograph apparatus and turned down to a thickness of 0.25 cm. Three minutes later it is tested on the alveograph which is kept at a constant temperature of 30°C.

A MATHEMATICAL ANALYSIS OF THE ALVEOGRAPH

One of the main objectives in this research is to determine whether the alveograph is suitable for the study of structural relaxation in bromated and unbromated doughs by a method analogous to that developed by Dempster et al (7) for the extensograph. It is necessary to show that a group of alveograms may be compared to each other at a constant sample deformation.

This section of the thesis is devoted to a mathematical analysis of the dough bubble, a study of the expansion process and the significance of the pressure recorded on an alveogram. An investigation of these factors will disclose whether or not a group of alveograms may be compared to each other at constant sample deformation.

The first factor which must be considered in this analysis is the geometry of the alveograph bubble. To understand the expansion of a dough bubble it is imperative to know the dimensions of different sizes of dough bubbles which can be inflated on the alveograph. These dimensions include the volumes and areas of dough bubbles.

The second step is to examine more closely the actual process of expansion of the dough membrane on the alveograph. This examination will include such variables as the change in volume and the area of the dough bubble with time. From an investigation of the variation of the volume and area of a dough bubble with time, a calculation of the variation of dough membrane thickness during the expansion process can be obtained. The variation of membrane thickness is an important factor which must be considered in the pressure changes which take

place during bubble expansion.

An alveogram is a graphical representation of the pressure changes taking place in the expansion of a dough membrane. Because the pressure is a fundamental measurement made on the alveograph, it is necessary to determine its exact significance in relation to the other variables which include volume and area of the bubble but more especially the thickness of dough membrane and the time variable.

The study of the geometry of the alveograph bubble, the bubble expansion process and the pressure variation during expansion serves as a basis for the construction of relaxation curves which will be described in the concluding portion of this section.

Geometry of the Alveograph Bubble

The alveograph bubble starts from a disc of dough 2.5 mm. in thickness and 5.5. cm. in diameter. As the bubble is raised, its height above the plate increases but the base remains the same since it is fixed by the dimensions of the apparatus. The alveograph bubble is actually a part of a sphere or in other words a spherical section.

Figure 6 shows four spheres on a constant base plate which illustrate the geometrical configurations as a bubble is blown on the alveograph from the early stage of the process when a small spherical section is above the base plate to a later stage when a large spherical section protrudes above the base plate. The diagram shows that a small spherical section above the base plate is part of a sphere having a large radius. As the size of the spherical section above the base plate increases, the radii

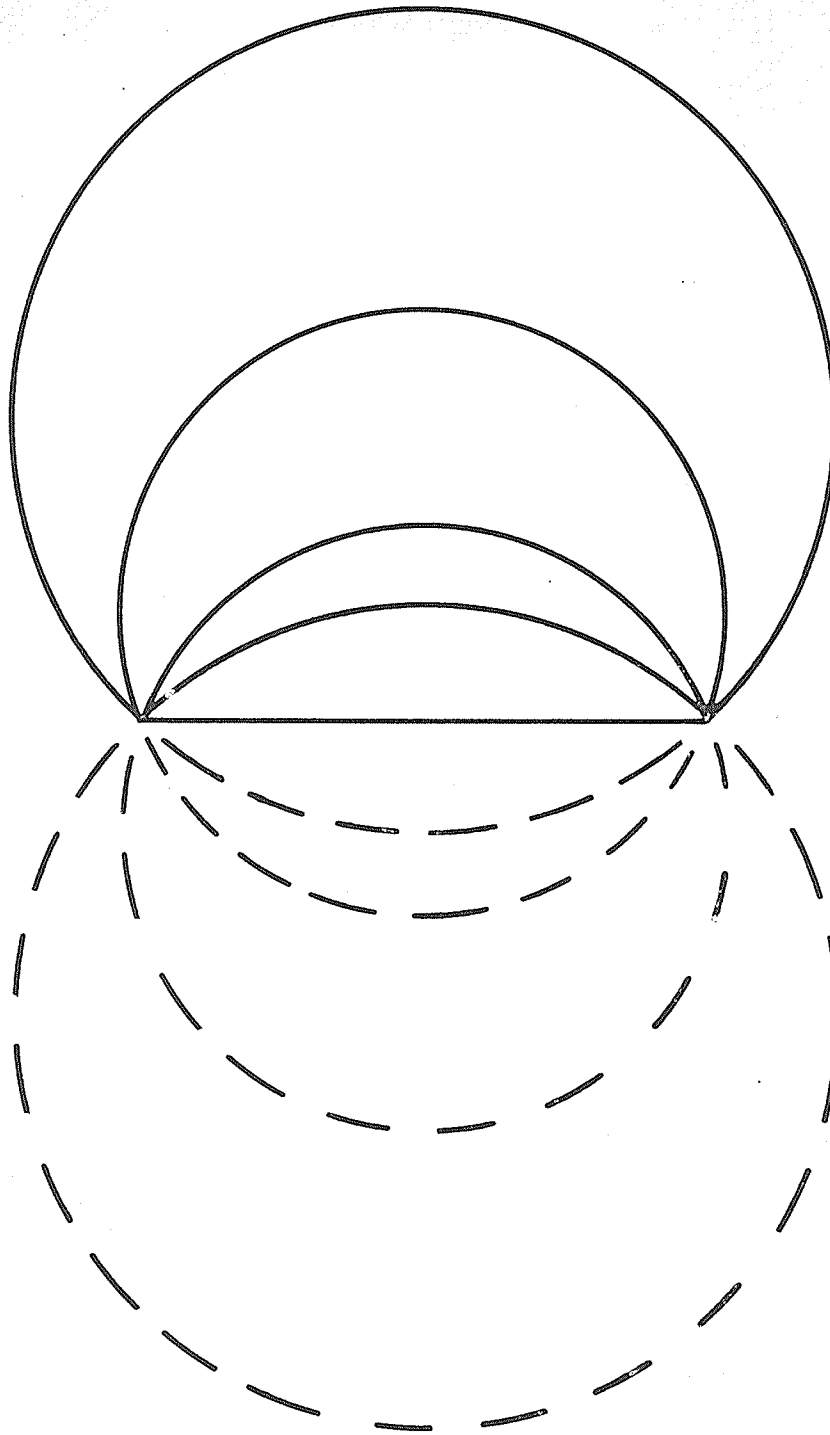


Fig. 6

Four spheres illustrating the geometrical configurations as a bubble is blown on the alveograph.

of corresponding spheres becomes smaller and smaller until the radius of the sphere becomes equal to the radius of the base plate. The radius then begins to increase again for larger spherical sections above the base plate.

The radius is a fundamental measurement in the calculation of volumes and areas of spheres and spherical sections. However the radius of a sphere cannot be measured directly and consequently it is better expressed in terms of the other geometrical factors which may be measured directly as illustrated in Fig. 7. From this diagram the height of the spherical section above the alveograph base plate is represented by X . The perpendicular distance of the base plate to the centre of the sphere is represented by $r - X$. The radius of the base plate 5.52/2 cm. Therefore by use of Pythagoras' theorem we have the following relation.

$$r^2 = \left(\frac{5.52}{2}\right)^2 + (r - X)^2$$

$$r = \frac{7.62 + X^2}{2 X}$$

For each distance X chosen arbitrarily a corresponding radius can be calculated. The heights of the spherical sections were chosen to include all sizes of bubbles which are of practical interest in reference to the alveograph. The corresponding radii for these X values were found to be in the range of from 2.7 to 7.9 cm. By choosing arbitrary heights of spherical sections the corresponding

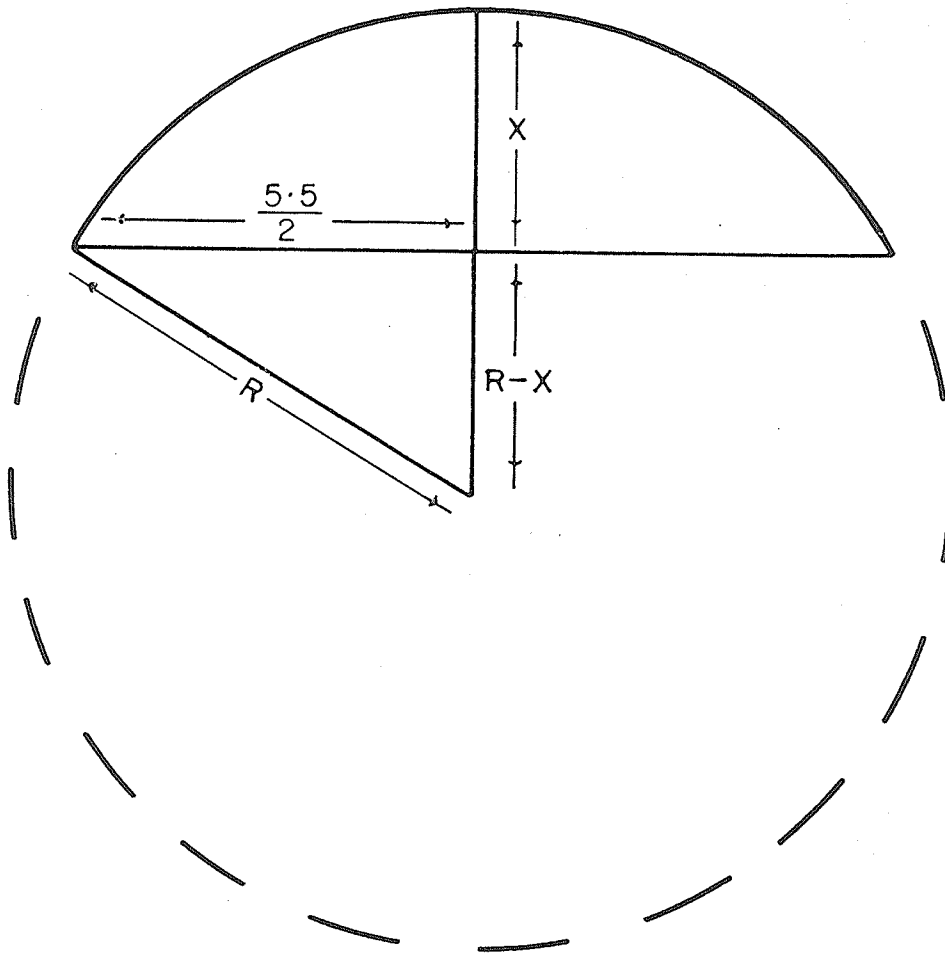


Fig. 7

A diagram showing the geometrical consideration necessary for the calculation of the radius of a sphere from the height of a spherical section above the alveograph base plate.

sphere radii have been calculated and will be used in the evaluation of areas and volumes of spheres.

Table I gives values for heights of spherical sections which have corresponding radii ranging from 2.77 cm. to 7.87 cm. The total volumes and areas corresponding to these radii are given in Table I. These areas and volumes were calculated from the formulae:

$$(1) S = 4 \pi r^2$$

where S = total area of a sphere

$$(2) V = 4/3 \pi r^3$$

where V = total volume of the bubble

The evaluation of the area and volume of a dough bubble which has been inflated on the alveograph may be described by referring to Fig. 6. In this figure the section of the sphere above the base plate (solid line) represents the dough bubble. The other section of the sphere (dotted lines) is shown below the base plate. A whole series of bubbles of varying sizes are thus built up on a constant base plate.

There are two methods which must be used to calculate the area and volume of a bubble namely: (a) for bubbles having a radius smaller than the radius of the base plate, the area and volume may be calculated directly, (b) for bubbles having a radius larger than the radius of the base plate the areas and volumes are obtained by subtracting the area or volume of the spherical section below

TABLE I

Total Areas, Volumes and Radii of Spheres Corresponding to Spherical Sections of Constant Base and Varying Heights.

Height of Spherical Section = X	Radius of Sphere = r	Total Area of Sphere = S	Total Volume of Sphere = V
cm.	cm.	cm ² .	cm ³ .
0.50	7.87	778.5	2048.
0.60	6.65	555.6	1235.
0.62	6.45	522.9	1127.
0.66	6.09	466.3	945.0
0.68	5.94	443.7	880.3
0.70	5.44	372.1	676.0
0.80	5.16	334.4	576.9
0.90	4.68	275.3	430.5
1.00	4.31	233.5	336.3
1.20	3.78	179.8	226.8
1.30	3.58	160.9	192.6
1.50	3.29	136.0	113.9
2.00	2.91	106.5	103.5
2.50	2.77	96.4	89.2

TABLE II

Areas, Volumes and Radii of Small Spherical Sections of Constant Base and Varying Height.

Height of Section = X	Radius of Sphere = r	Area of Small Spherical Section = S_s	Volume of Small Spherical Section = V_s
cm.	cm.	cm ² .	cm ³ .
0.50	7.87	24.6	6.0
0.60	6.65	25.0	7.2
0.62	6.45	25.1	7.4
0.66	6.09	25.2	7.9
0.68	5.94	25.3	8.7
0.70	5.44	25.4	8.4
0.80	5.16	25.9	9.9
0.90	4.68	26.4	11.1
1.00	4.31	27.0	12.4
1.20	3.78	28.4	15.1
1.30	3.58	29.2	16.7
1.50	3.29	31.7	19.6
2.00	2.91	38.0	27.9
2.50	2.77	43.5	37.8

the base plate from the total area or volume of the sphere. The following describes the calculations of the volumes and areas of spherical bubbles using the two methods.

Table II gives the values for the heights of the spherical sections and the corresponding radii of the spheres for each section. The values for these areas and volumes of the small spherical sections are given in this table.

The volume of small spherical sections, Table II column (4), is obtained from the formula given in the Handbook of Physics and Chemistry (14).

$$V_s = 1/6 \pi (x^2 + 3a^2)$$

where V_s = volume of small spherical section

a = radius of the base of the segment.

The area of a small spherical section, Table II, column (3) is given by the formula:

$$S_s = 2 \pi r X$$

where S_s = area of small spherical section

r = radius of sphere

X = height of spherical section.

The radius of a sphere cannot be directly determined and consequently it is redefined in terms of the length of the base of the spherical section and the height of the spherical section. Expressing r in terms of these factors the following expression for the area of the

spherical section is obtained:

$$S_s = \pi/4 (4 X^2 + C^2)$$

where S_s = the area of the small spherical section

C = the length of base of spherical section.

The values of the areas of small spherical sections are given in Table III. Column (1) of the table gives the radii of spheres between the range of 2.77 cm. to 7.87 cm. The areas of small spherical sections and the total areas of the spheres corresponding to these radii are given in columns (2) and (3) of the table. The area of the large spherical section S_L (area of a bubble) is given in column (4) of the table. The area of a bubble or large spherical section is obtained by subtracting the area of the small spherical section S_s from the total area of the sphere S . Thus $S_L = S - S_s$.

Table IV gives the volume of large spherical sections. Column (1) gives the radii of spheres from 2.77 cm. to 7.87 cm. The total volumes V of the spheres and the volumes of the small spherical sections V_s corresponding to these radii are shown in columns (2) and (3) of this Table. The volumes of the large spherical sections (volumes of bubbles) with corresponding radii from 2.77 to 7.87 cm. are given in column (4).

The volume of a large spherical section is obtained by subtracting the volume of a small spherical section from the total volume of a sphere i.e. $V_L = V - V_s$.

TABLE III

Area of Large Spherical Sections of Radius 2.77 cm. to 7.87 cm.

Radius of Sphere = r	Total Area of Sphere = S	Area of Small Spherical Section = S_s	Area of Large Spherical Section = S_L
cm.	cm ² .	cm ² .	cm ² .
2.77	96.4	43.5	52.9
2.91	106.5	38	68.5
3.29	136.0	31.0	104.3
3.58	160.9	37.1	123.8
3.78	179.8	36.1	143.7
4.31	233.5	27.0	206.5
4.68	275.3	33.6	241.7
5.16	334.4	32.9	301.5
5.44	372.1	32.3	339.8
5.94	443.7	32.2	411.5
6.09	466.3	32.1	434.0
6.45	522.9	31.9	491.0
6.65	555.6	31.8	523.8
7.87	778.5	24.6	753.9

TABLE IV

Volume of Large Spherical Sections of Radius 2.77 cm. to Radius 7.87 cm.

Radius of Sphere = r	Total Volume of Sphere = V	Volume of Small Spherical Section = V_s	Volume of Large Spherical Section = V_L
cm.	cm ³ .	cm ³ .	cm ³ .
2.77	89.3	37.8	51.5
2.91	103.5	27.9	75.6
3.29	148.6	19.6	129.06
3.58	192.6	16.7	175.9
3.78	226.8	15.1	211.0
4.31	336.3	12.4	323.9
4.68	430.5	11.1	419.4
5.16	576.9	9.87	567.0
5.44	676.0	8.41	667.6
5.94	880.3	8.16	872.1
6.09	945.0	7.92	937.1
6.45	1127.	7.44	1119.
6.65	1235.	7.20	1228.
7.87	2048.	6.01	2042.



Variation in Volume, Area and Dough Membrane Thickness

During the Expansion of a Dough Bubble in the Alveograph.

The variables involved in the process of expansion of a dough membrane include volume of a bubble, area of a bubble, the thickness of the dough membrane and time. This section will consider time-dependent changes that take place with these variables during this process and how the relationship between time, volume and area of the dough bubble may be utilized to determine the membrane thickness at any instant during the expansion process.

One of the most important of these relationships is the volume of the dough bubble vs. time. The most practical way of measuring the volume of the bubble is by measuring the amount of air displaced by the water in the cylinder during the expansion process. It is reasonable to assume that the volume of air displaced is equal to the volume of water which enters the plastic cylinder. To verify this relationship the actual pressure changes taking place during the expansion process must be considered (a) using a membrane in the alveograph apparatus (b) not using a membrane in the alveograph apparatus.

A difference between the volume of air and volume of water may arise from the compression of air within the dough bubble. This air compression equals the atmospheric pressure plus the air pressure within the bubble. The estimated maximum pressure within the bubble which can be recorded in an alveogram is equivalent to 159 mm. H₂O or 11.7 mm. Hg. Therefore the total maximum pressure is $\approx 740 + 11.7 = 751.7$ mm. Hg.

Now taking for example a volume of water of 100 ml. the volume of the bubble corresponding to this will be

$$100 \times \frac{740}{751.7} = 98.4 \text{ cc.}$$

Thus for the maximum case there is only a difference of 1.6% in the two readings. For the majority of alveograms the difference will be one half or less of the value for the maximum case. This treatment thus shows that for all practical purposes the volume of the bubble may be taken as equal to the volume of water entering the cylinder.

The rate of flow with and without a dough patty in the apparatus was tested to ascertain whether the back air pressure caused by the membrane had an appreciable effect on the rate of water flow. Dough patties used for testing the water flow with a membrane were given long and short rest periods (recording high and low pressures on the kymograph).

The method used to obtain the values for volume and time for the graph is described as follows: Six stop watches are started instantaneously when the water begins to flow in the burette. One watch is stopped after each 100 cc. water has flowed into the burette. In this way the time in seconds is recorded for every 100 cc. water which flows into the burette up to a value of 800 cc. Volume-time values were recorded for three arbitrarily chosen rates of flow namely 25.1, 19.0 and 14.8 ml./sec.

A graph (Fig. 8) was made for the three rates of flow mentioned. Examination of the plots reveals a linear relationship passing through the origin indicating that the rate of flow is directly proportional to time in this range. The open circle plots on the graph show the rate of

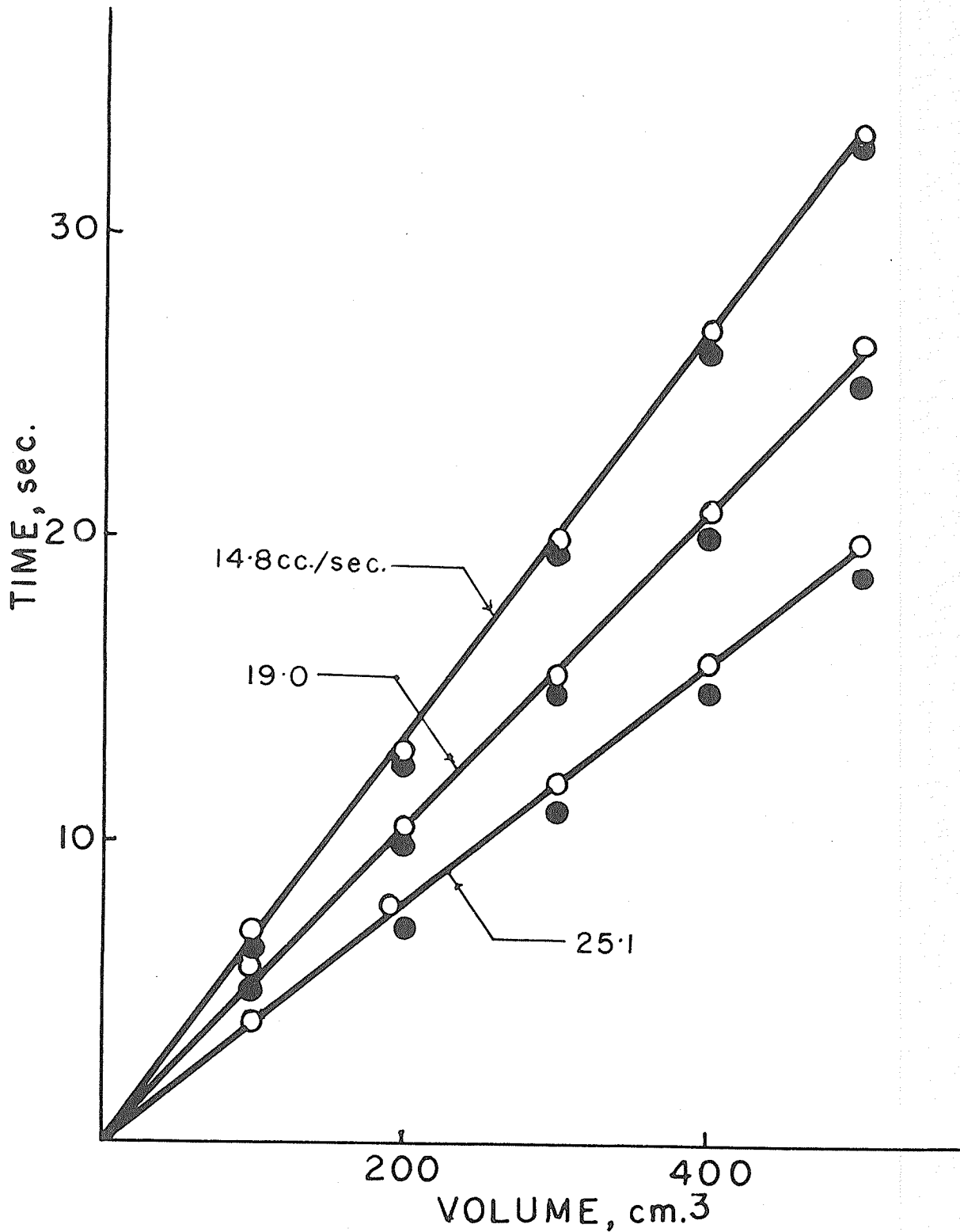


Fig. 8

Volume of air displaced in burette vs. time relationship for the three rates of water flow 25.1, 19.0 and 14.8 ml./sec.

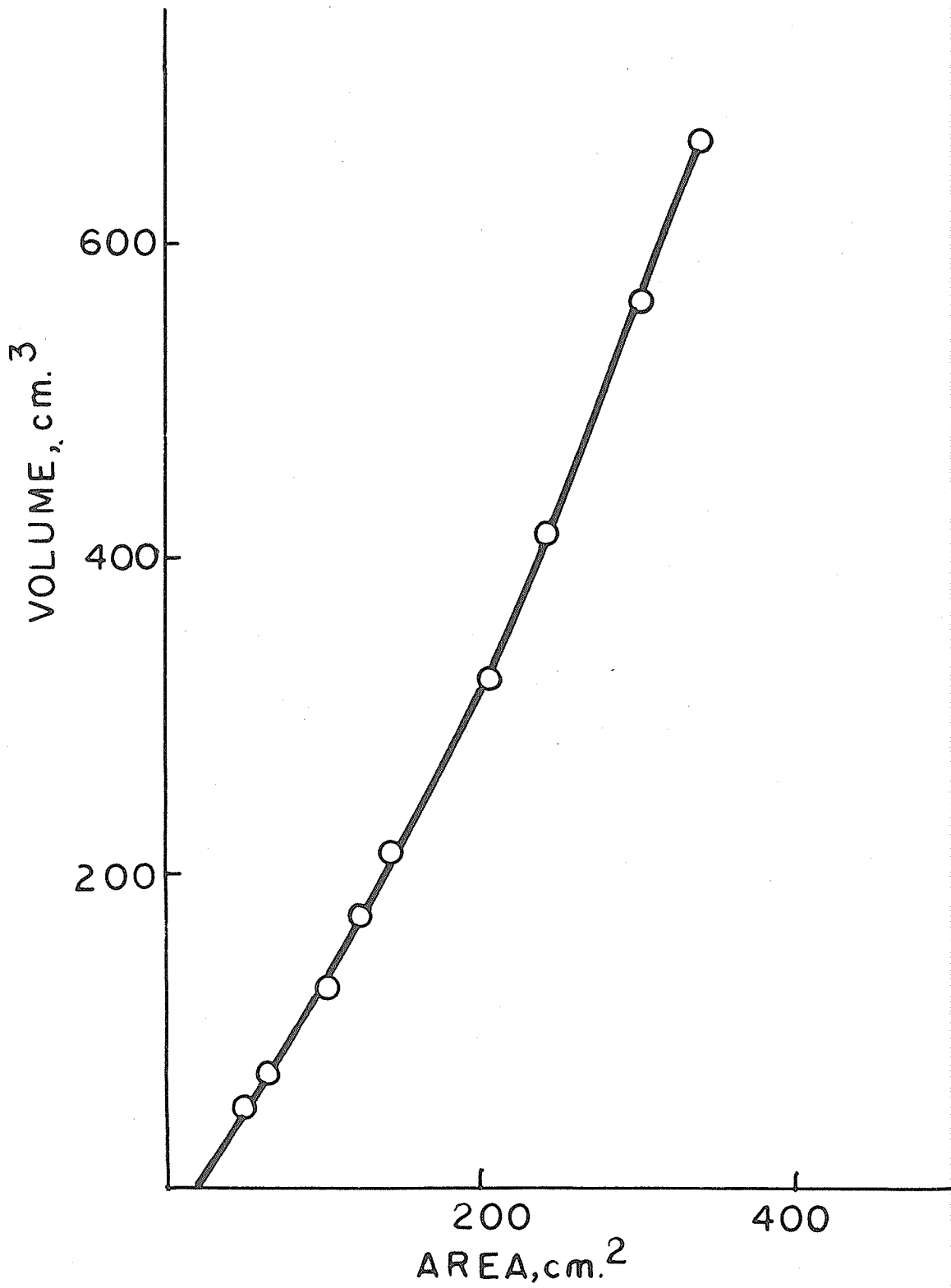


Fig. 9

A plot showing the relationships between volumes and areas of alveograph bubbles.

flow without a membrane and the closed circle dots show the rate of flow with a membrane. The difference, as shown by the graph, between rate of flow with and without a membrane is negligible.

Therefore it has been proved through direct calculation of the air compression and experimental results obtained on the resistance of a dough membrane to air flow with the alveograph that the volume of water recorded at any time during the expansion process may be used as a measure of the volume of the bubble.

The area of a bubble at any specific time can be determined by plotting the areas of the bubbles already calculated against the volumes of the dough bubbles. Then knowing the volume of a dough bubble at any time the area can be found by reading it from the plot of volume vs. area of bubble. This plot is shown in Fig. 9.

It is now possible to construct a graph of area vs. time (Fig. 10) from which the area of the bubble at any specific time will be obtained directly. This relation is important for calculating the thickness of the dough membrane at any time during the expansion.

An alveogram is a record of the resistance offered by a dough membrane during the expansion of a dough bubble. From first principles it can be assumed that this resistance is a function not only of the pressure built up within the bubble but also of the thickness of the dough membrane. It is therefore necessary in alveogram analysis to be able to calculate the thickness of a dough membrane at any time during the expansion process and to refer the pressure to unit thickness.

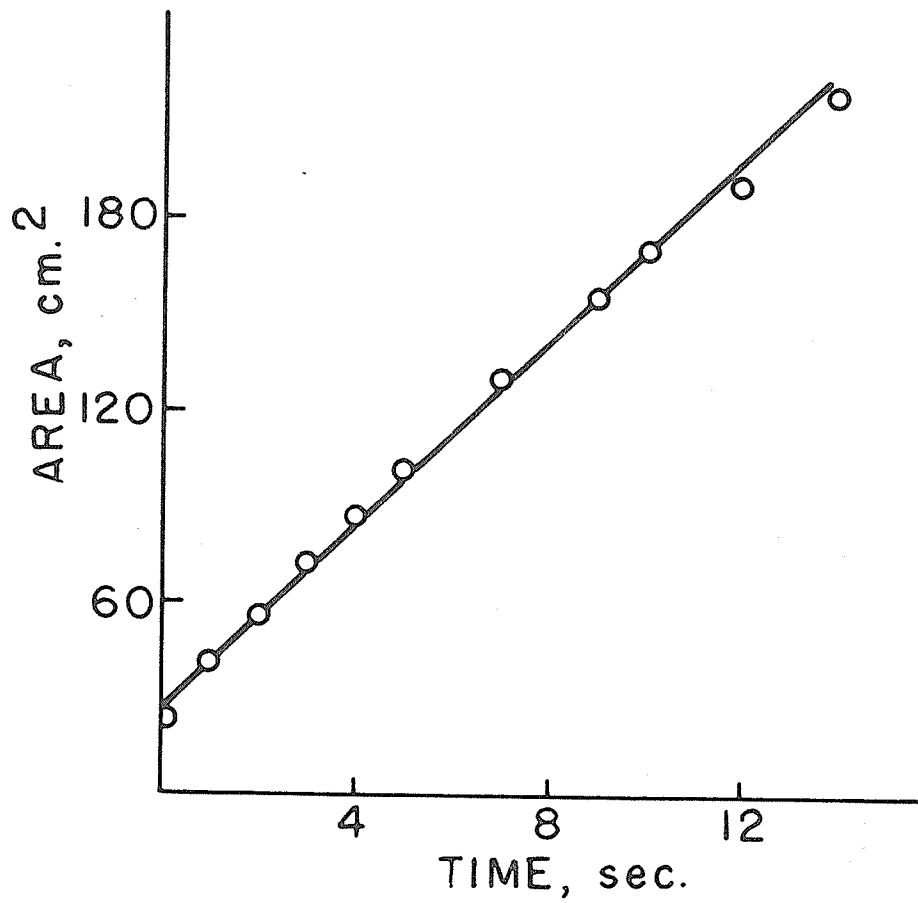


Fig. 10

A plot of area of an alveograph bubble vs. time.

This variable is determined according to the following manner: An arbitrary distance is selected along the base of the alveogram. This distance corresponds to a definite time which can be determined, knowing the velocity of the revolving kymograph drum. Referring to the area vs. time plot Fig. 10, the area of the bubble corresponding to this particular time can be read from the graph.

The volume of the original dough patty before expansion divided by the area of the bubble at the specific time used, gives the thickness of the membrane at that particular time. The volume of the dough patty before expansion can be found from the relation:

$$V = \pi r^2 h$$

$$V = \pi \times \left(\frac{5.51}{2}\right)^2 \times 0.25 = 5.94 \text{ cm.}^3$$

where V = volume of patty

r = radius of patty

h = height of patty

Therefore the thickness of a dough membrane at time "t" =

$$\frac{5.94}{\text{area of bubble at time "t"}}$$

Table V summarizes all data of area, volume and thickness of dough membranes made in this section for the three rates of flow i.e. 25.1, 19.0 and 14.9 ml./sec. Column (1) gives the arbitrarily chosen distances along the base of the alveogram and column (2) gives the time in seconds corresponding to these distances. Columns (3) and (4) give the areas and volumes of dough bubbles. Column (5) gives the thickness of dough membrane in mm.

TABLE V

Area, Volume and Thickness of Dough Bubbles Corresponding to Alveogram Base Lengths from 0.118 to 8.83 cm.

Distance Along Base of Alveogram	Time	Area of Dough Bubble	Volume of Dough Bubble	Thickness of Dough Membrane
cm.	<u>25 ml./sec.</u>	cm. ²	cm. ³	mm.
0.118	0.2 sec.	28	5	2.12
0.295	0.5	32	12	1.86
0.59	1	42	25	1.41
1.10	2	57	50	1.04
1.77	3	73	75	0.81
2.36	4	87	100	0.68
2.95	5	103	125	0.58
4.22	7	132	175	0.45
5.33	9	157	225	0.38
5.89	10	170	250	0.35
7.26	12	190	300	0.31
8.46	14	218	350	0.27
8.83	15	228	375	0.26
<u>19.0 ml./sec.</u>				
0.118	0.2	27	4	2.20
0.295	0.5	30	7	1.98
0.59	1	37	17	1.61
1.10	2	45	29	1.32
1.77	3	69	55	0.99
2.36	4	73	75	0.81
2.95	5	85	94	0.70
4.22	7	107	131	0.56
5.33	9	128	170	0.46
5.89	10	142	189	0.42
7.26	12	157	225	0.38
8.46	14	178	265	0.33
8.83	15	183	283	0.32
<u>14.8 ml./sec.</u>				
0.118	0.2	26	2	2.28
0.295	0.5	28	6	2.12
0.59	1	34	14	1.75
1.10	2	45	29	1.32
1.77	3	54	44	1.10
2.36	4	64	58	0.93
2.95	5	73	73	0.81
4.22	7	97	117	0.61
5.33	9	107	132	0.56
5.89	10	117	147	0.51
7.26	12	133	177	0.45
8.46	14	143	207	0.42
8.83	15	155	221	0.38

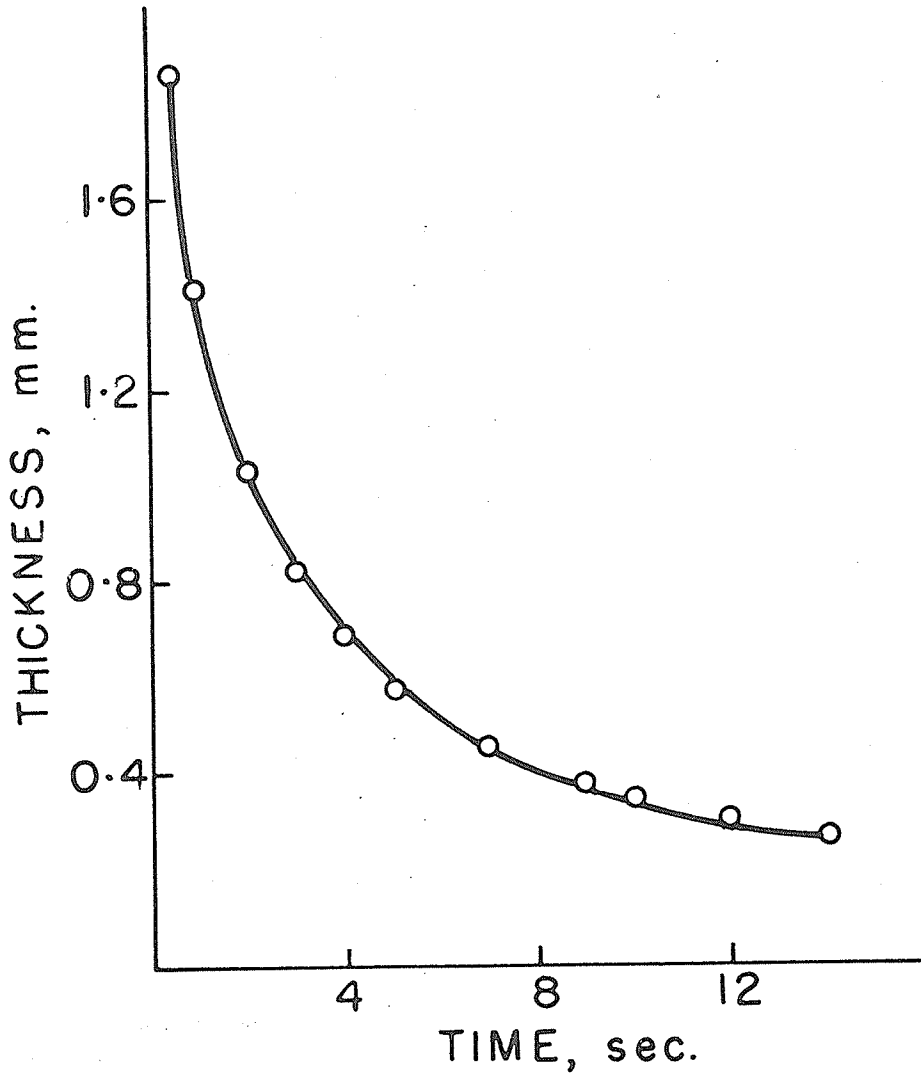


Fig. 11

Thickness of dough membrane vs. time.

It is now possible to draw a graph of thickness of membrane vs. time Fig. 11. From this graph, the thickness of a dough membrane can be determined for any arbitrarily chosen time.

The Significance of Pressure in the Expansion of
a Dough Bubble in the Alveograph.

An alveogram measures the pressure changes taking place during the expansion of a dough bubble. Since the pressure is a fundamental measurement made on the alveograph it is necessary to investigate the significance of the pressure in relation to the geometry of the alveograph bubble during the expansion process.

It is now possible to refer the pressure within the bubble to unit thickness at any time during the expansion process. An ordinary alveogram can be transformed in terms of resistance of the dough (pressure/unit thickness) P/d vs. time. The transformation of an alveogram is done in the following way: the pressures are recorded from the alveogram for the distances along the base used in the calculation of the membrane thickness. Knowing the thickness of membrane corresponding to these distances the resistance ($\frac{P}{d}$) may be determined. Figure 12 shows the resistance vs. time plot for rest periods of 5, 7, 10 and 20 min. under the conditions of 1 hr. reaction time and 0 p.p.m. $KBrO_3$.

Examination of the resistance vs. time plot reveals that the resistance caused by the dough membrane rises continually until membrane rupture. The plot clearly shows that the maximum displayed on an ordinary alveogram has no significant meaning.

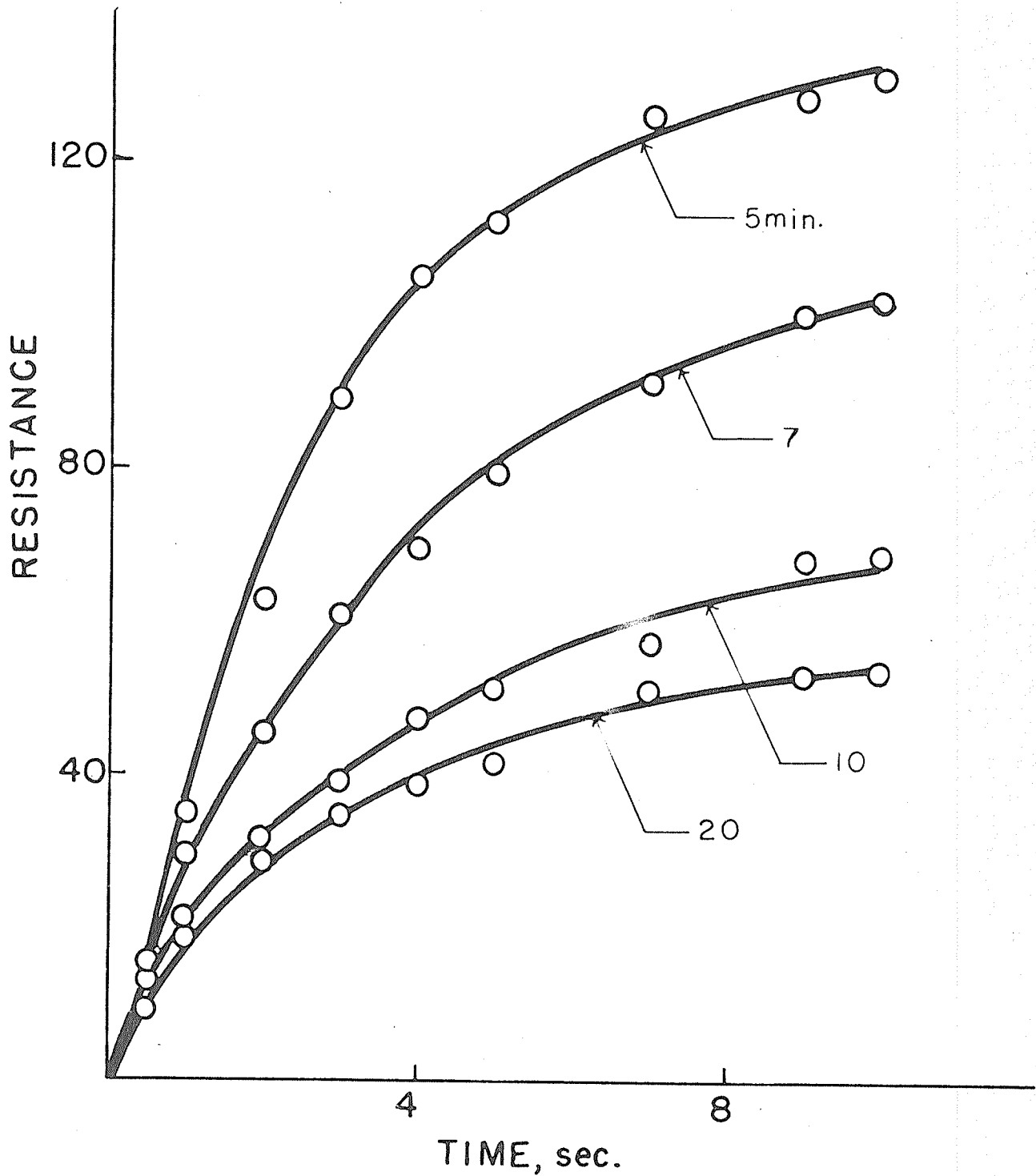


Fig. 12

A plot of the transformation of a typical alveogram taking into consideration the thickness of a dough membrane.

The resistance vs. time plot does not reveal whether this set of curves is a family of curves. It is necessary to test these curves to ascertain whether or not they belong to one family in order to be able to make a further analysis of alveograms.

Certain standard plots were tried but the only significant plot which could be used to establish whether a set of alveograms belonged to one family of curves was the resistance x time vs. time relationship. Figure 13 shows this plot for 0 p.p.m. KBrO_3 and 1 hr. reaction time and the partially straight line plots obtained suggest there is a very approximate reciprocal relationship between the resistance and the time.

It was decided to substitute the area of the bubble for the time factor, such that resistance x area would be plotted against area, for two reasons: (1) the time factor does not take into consideration the original area of the dough patty before expansion takes place while the resistance x area vs. area plot accounts for the original area of the dough patty shown by a definite intercept on the x-axis. (2) The rate of change in area of the bubble is more fundamental to the expansion process than time due to the fact the area is inversely proportional to the thickness of the membrane. Figure 14 shows a set of curves plotted for 1 hr. reaction time and 0 p.p.m. KBrO_3 and the linearity of these plots reveals that these curves all belong to one definite family. The transformations made on alveogram curves cover a range of 0.59 to 8.83 cm. along

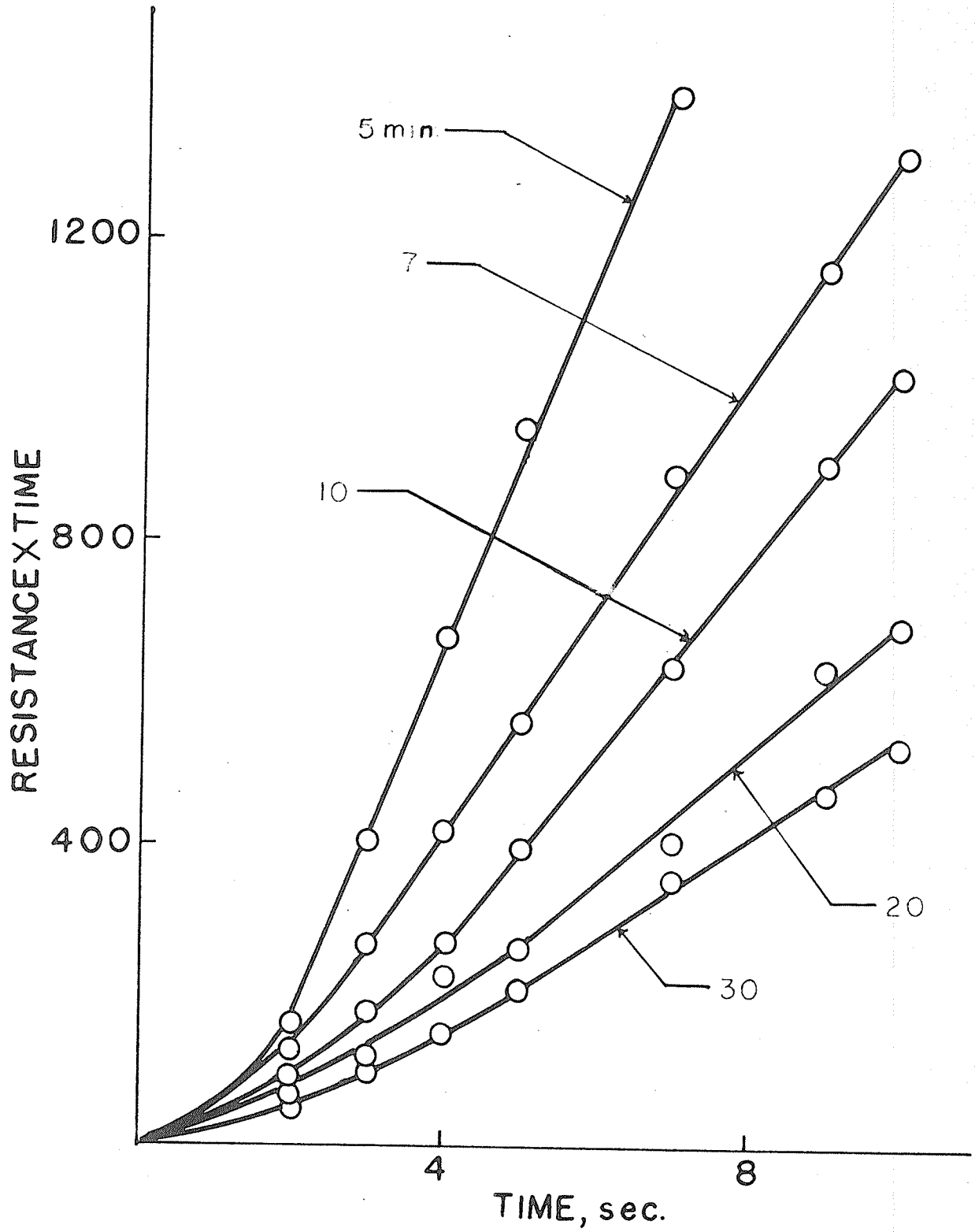


Fig. 13

The resistance to extension x time vs. time transformation of a typical alveogram.

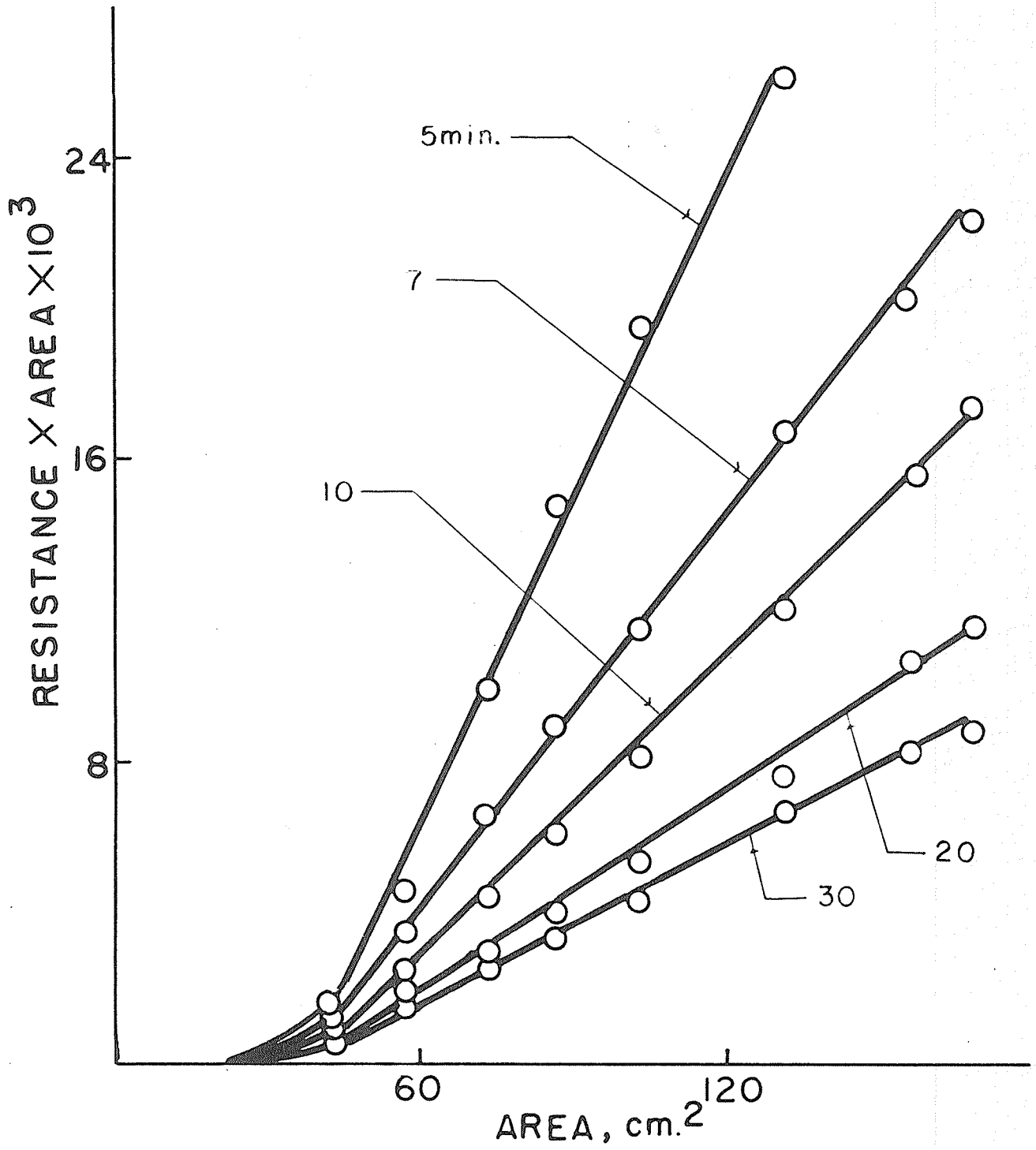


Fig. 14

The area x resistance to extension vs. area transformation of a typical alveogram.

the base of the alveogram. This range sufficiently covers the most important section of the alveogram and for all practical purposes the remainder of the curve may be neglected.

The linearity of these plots holds over a large portion of the curve with the exception of the short range in the vicinity of the intercept, i.e., at the beginning of the expansion process. This deviation from linearity might be attributed to two factors:

(a) The first stages of expansion are not uniform and it might be assumed that a symmetrical spherical section is not immediately formed at the beginning of expansion which might cause a deviation in the linearity of the curves.

(b) In the standard procedure a few cc. of air are injected under the patty to raise it off the base plate so that when expansion begins the patty will not stick to the base plate. This factor might cause a slight variation in the true pressure and consequently cause a small initial deviation in the linearity of the plots.

An understanding of the significance of the slope of these plots is necessary in making a fundamental alveogram analysis. The slope may be expressed as $\frac{\text{resistance} \times \text{area}}{\text{area}}$ or simply as resistance $(\frac{P}{d})$.

Thus it may be stated that the pressure measured at any point along the straight line plots is referred to unit thickness or in other words the resistance caused by 1 cm.² of dough membrane, 1 mm. in thickness. In the special case where $d = 1$ the slope may be determined directly in terms of pressure.

For the purpose of constructing a relaxation curve it is necessary to measure the resistance $\frac{P}{d}$ at only one point on the straight line plots. This arbitrarily chosen point corresponds to a certain definite thickness of dough membrane and all curves are analysed at this thickness. This may be chosen in two ways:

- (1) It may be chosen so that it will be equal to unity in which case the resistance may be designated directly in terms of pressure.
- (2) A constant thickness "d" may be chosen. A constant value of the thickness will not affect the relative magnitudes of the pressures recorded and thus for practical purposes the resistance may be measured directly at a fixed membrane thickness.

It is now possible to construct relaxation curves directly from alveograms by a method analogous to the method used by Dempster et al. (7) for extensogram analysis. A constant membrane thickness of 0.74 mm., corresponding to a distance of 2 cm. along the base of an alveogram, was arbitrarily chosen for alveogram analysis. The pressure recorded at this deformation is measured along the arc of the kymograph directly in terms of the height of the alveogram (mm.). To obtain a structural relaxation curve the pressure values obtained from a set of alveograms are plotted against rest period.

EXPERIMENTAL ALVEOGRAM DATA

This thesis may be divided essentially into two main sections. The first section involves the mathematical analysis of the alveogram leading ultimately to a method by which groups of alveograms can be analysed. The second section involves the application of the method of analysis to alveograms obtained under a wide variety of conditions.

The conditions under which doughs were tested include reaction times of 0, 1, 2, 3 and 4 hr., rest periods of 5, 7, 10, 20, 30 and 45 min., bromate concentrations of 0, 10 and 30 p.p.m. and rates of flow of 14.8, 19.0 and 25 ml./sec. A wide selection of alveograms was thus obtained which could be used for testing the validity of the method of analysis under many different sets of conditions.

Since the method for analysis of alveograms has already been described in the previous section it is now possible to show, from a wide selection of alveograms, that the study of the structural relaxation of bromated and unbromated doughs can be carried out in an analogous manner to that developed by Dempster et al in the analysis of extensograms. The applicability of the method proposed by Dempster et al to the alveograph is demonstrated by (a) proving the validity of the straight line resistance x area vs. area plots under varied conditions (b) the construction of structural relaxation curves and their hyperbolic characterization.

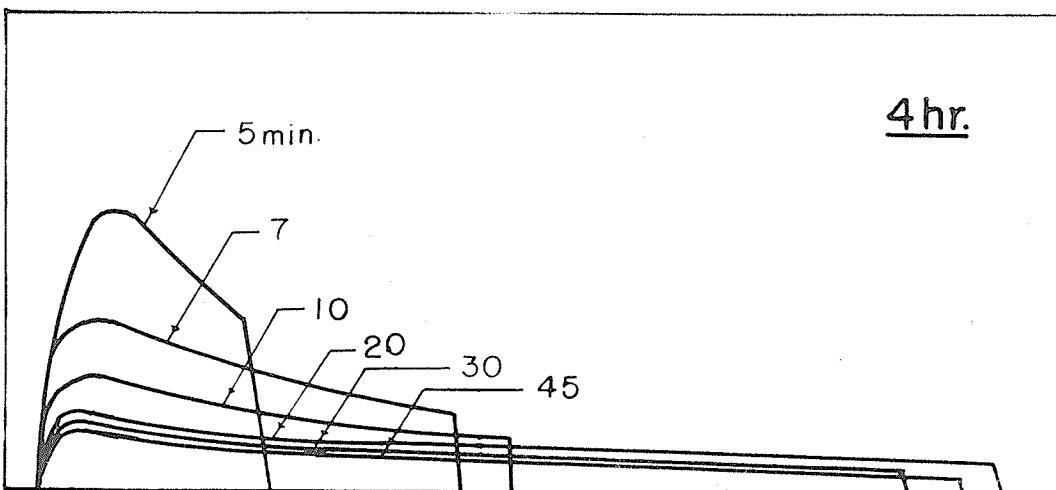
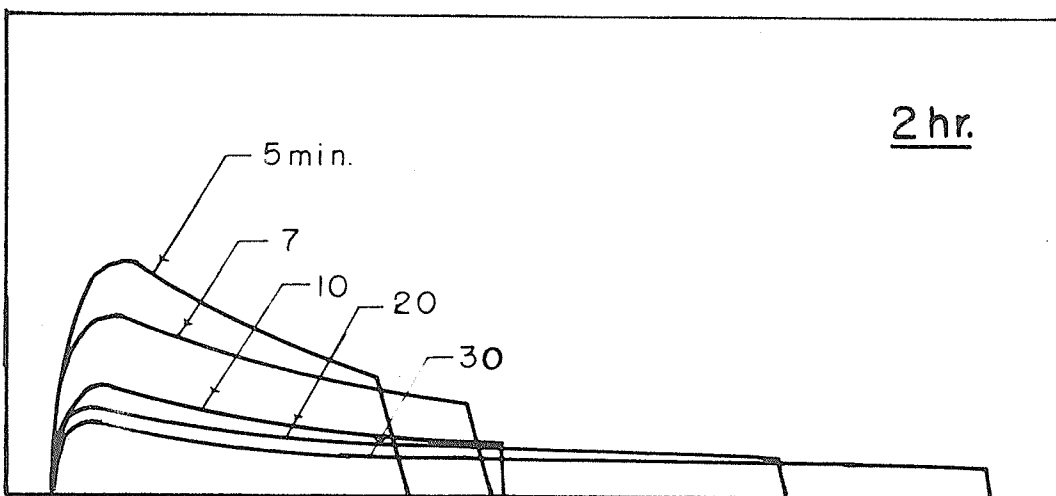
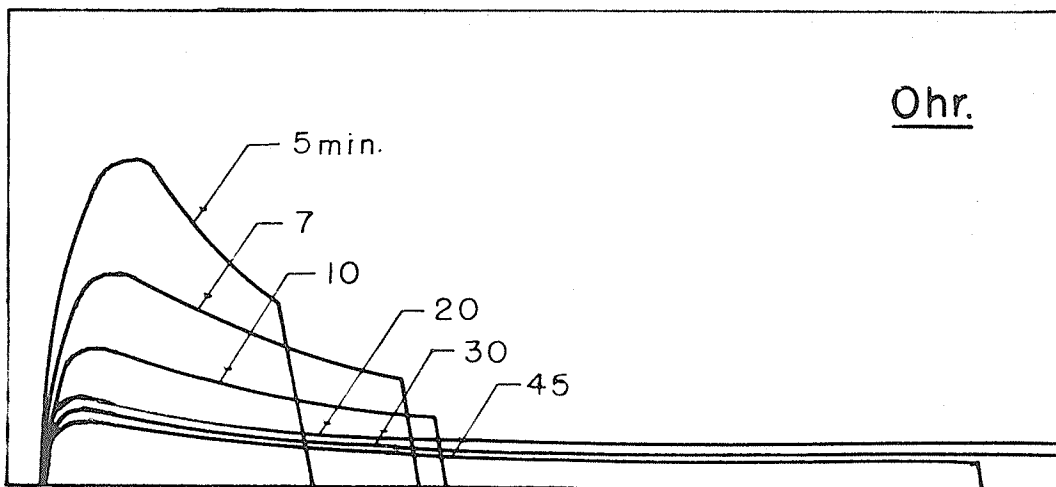


Fig. 15

Sets of alveograms for a variety of rest periods and reaction times and 0 p.p.m. potassium bromate.

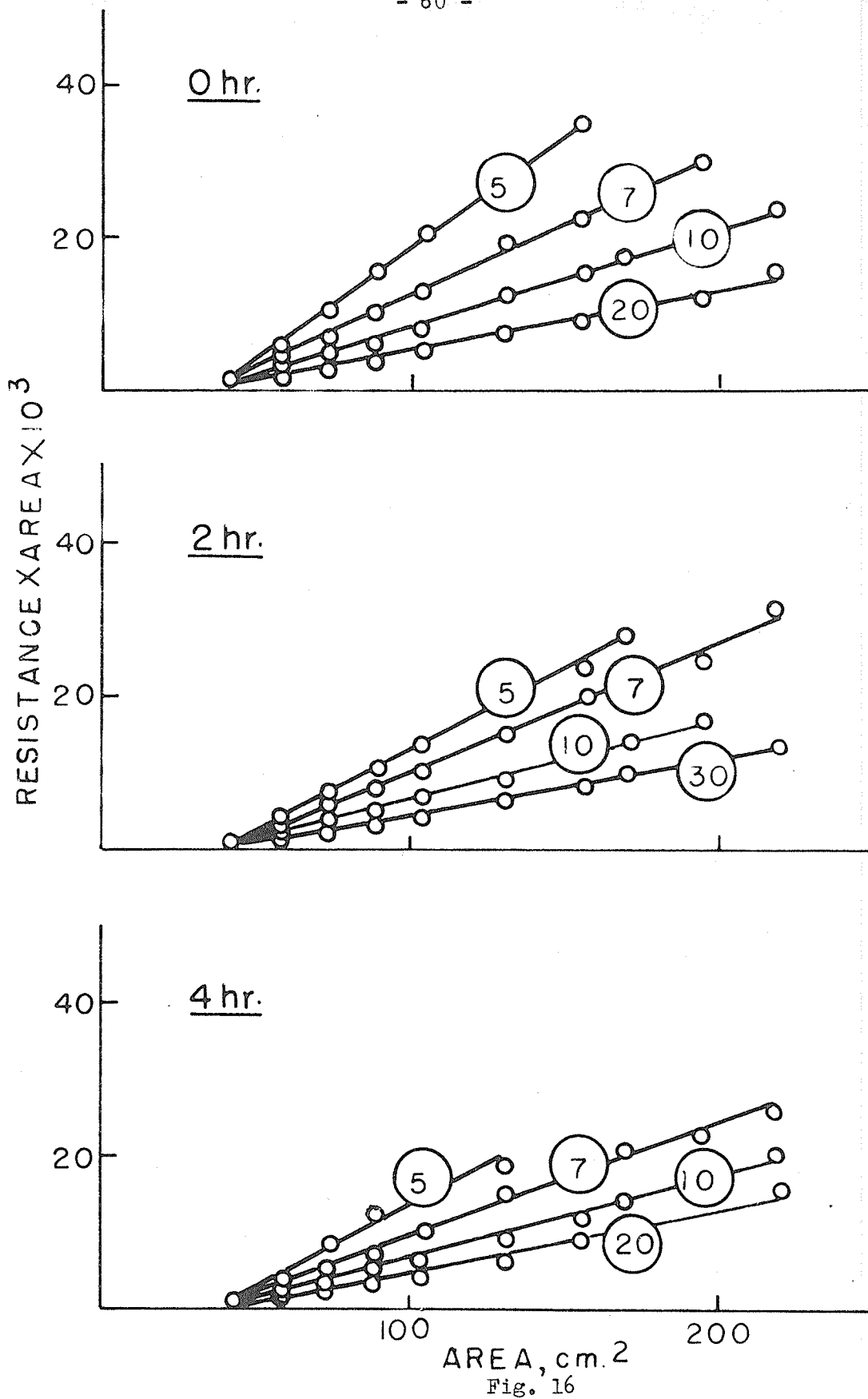


Fig. 16

Graphs illustrating the validity of the linear relationship of resistance x area vs. area corresponding to sets of alveograms in figure 15.

An examination of alveograms treated under many different conditions will also be carried out to determine the significance of the standard measurements which have been made on an alveogram. These measurements include the alveogram maximum, the square root of the volume of the dough bubble, extensibility (length of an alveogram), area under the alveogram curve and the work function.

Structural Relaxation in Bromated and Unbromated Doughs

A Study of the Resistance x Area vs. Area Plot Under a Wide Variety of Conditions. It has been shown in the previous section that the resistance that a dough bubble offers to extension is inversely proportional to the area of the bubble. The investigation of the structural relaxation process in dough is carried out under many different conditions including variations in rest period, reaction time and bromate concentration. It is therefore necessary to test the validity of the plot of resistance x area vs. area, showing that the resistance $\propto \frac{1}{\text{area}}$ under a wide variety of conditions by determining the goodness of fit of points on the graph.

Figures 15, 16, 17, 18 show sets of alveograms and the corresponding resistance x area vs. area transformations for dough samples treated under the following conditions, 0 and 10 p.p.m. KBrO_3 , 0, 2 and 4 hr. reaction time, and 5, 7, 10, 20 and 30 min. rest periods. The transformations cover the most important part of the alveogram curve (from 0.8 to 8.8 cm. along the base of the alveogram) and for all practical purposes the rest of the curve may be neglected.

An analysis of the data shows that all the graphs are linear with all points very close to the curves proving definitely that the area

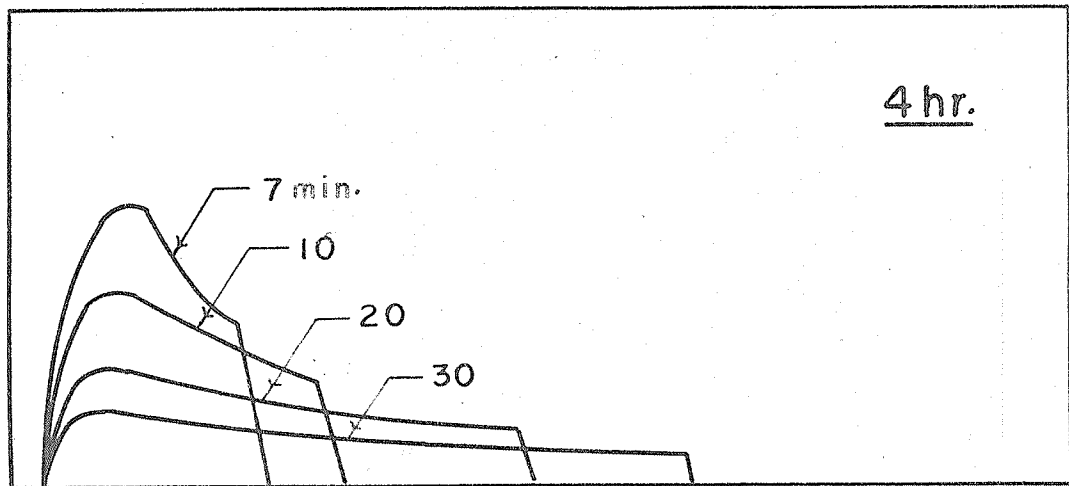
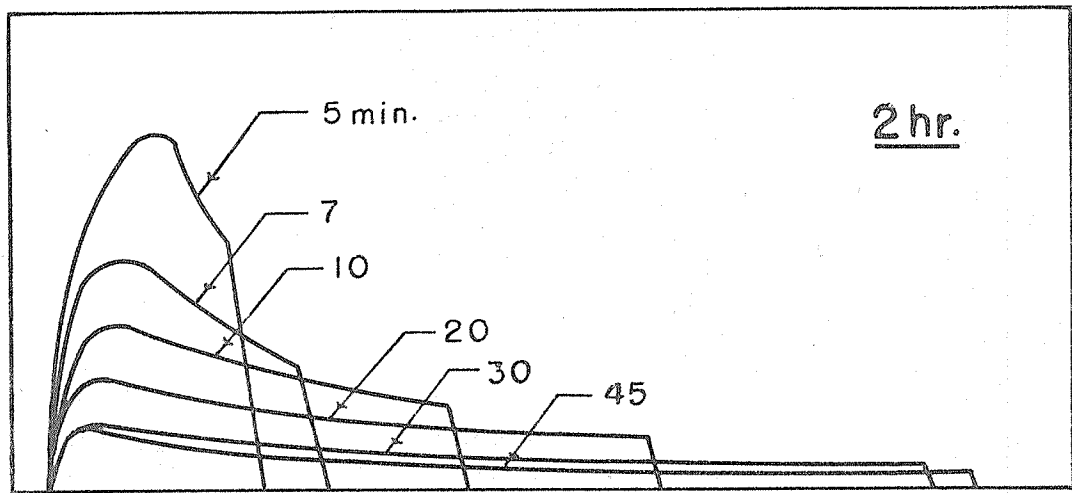
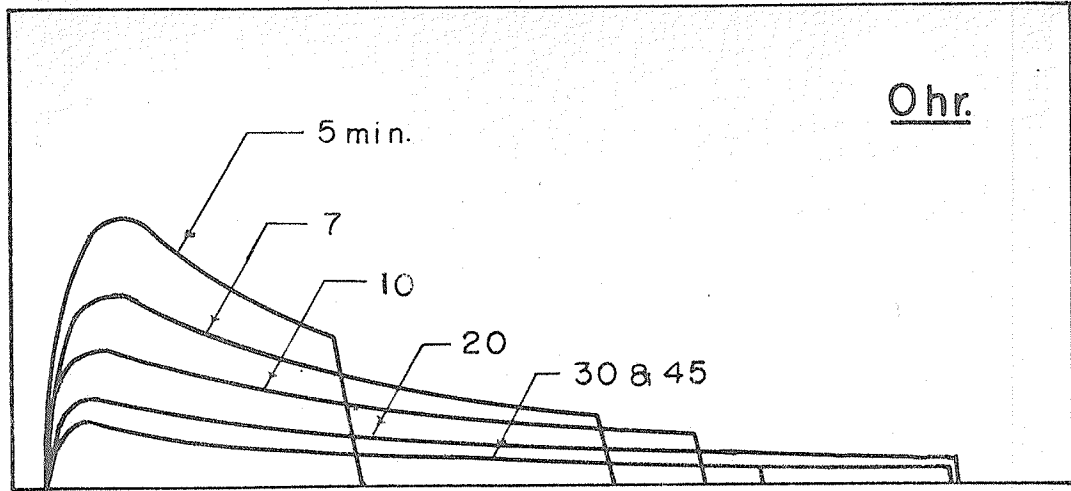


Fig. 17

Sets of alveograms for a variety of rest periods and reaction times and 10 p.p.m. potassium bromate.

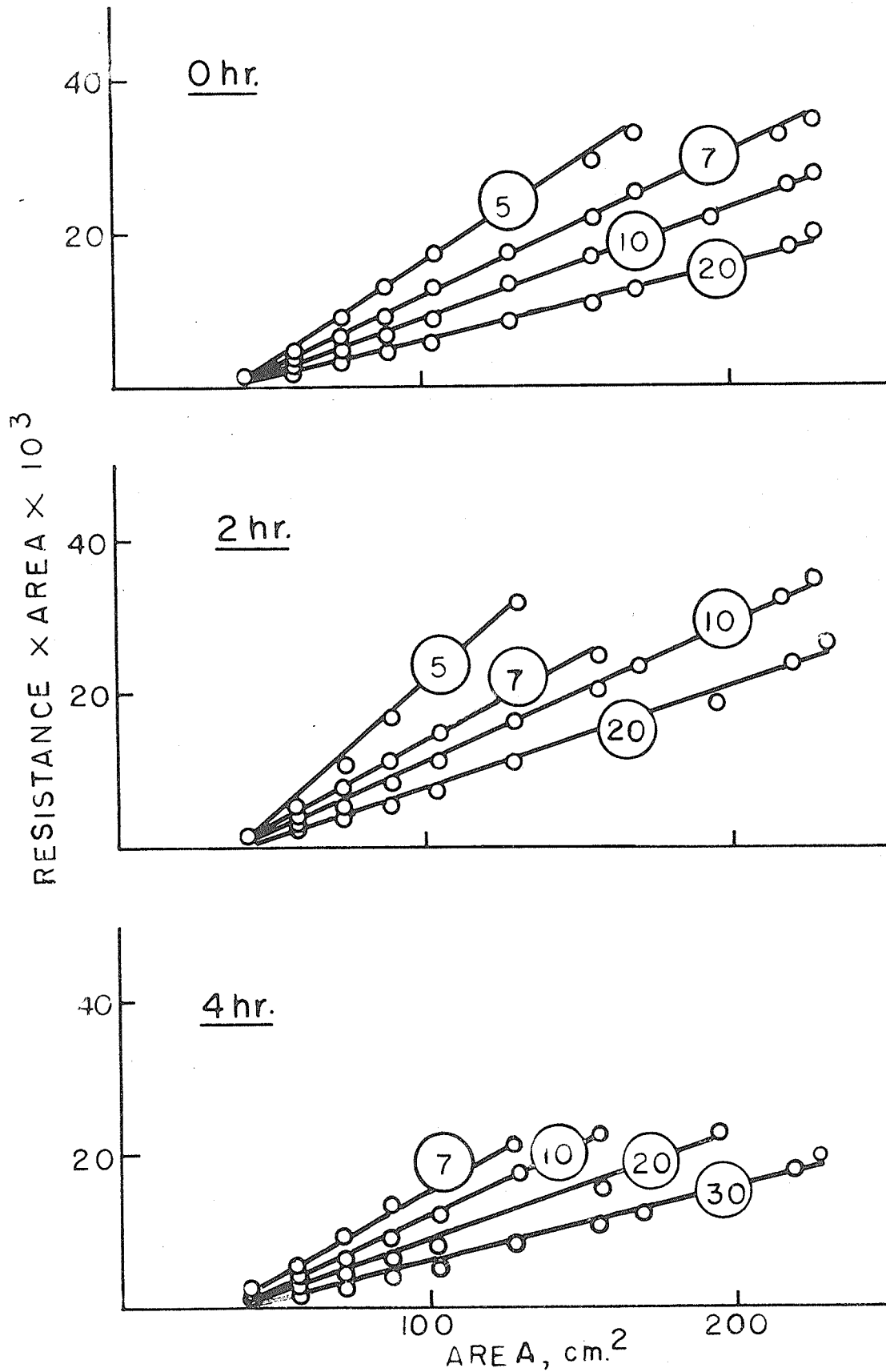


Fig. 18

Graphs illustrating the validity of the linear relationship of resistance x area vs. area corresponding to sets of alveograms in figure 17.

of the bubble is inversely proportional to the resistance under a wide variety of conditions. A direct consequence of this relationship is that a group of alveograms belongs to a definite family of curves for a wide selection of conditions. It can now be safely assumed that a set of alveograms are comparable to one another at a constant sample deformation.

Structural Relaxation Curves. The establishment of the fact that a set of alveograms are comparable at a constant sample extension makes it possible to study the structural relaxation process in bromated and unbromated doughs. The method used for this study is analogous to that used by Dempster et al (7) for the extensograph and has been described in a previous section.

Doughs were given reaction times of 0, 2 and 4 hr., rest periods of 5, 7, 10, 20, 30 and 45 min. and bromate concentrations of 0, 10 and 30 p.p.m. potassium bromate. The resistance was measured directly in terms of alveogram height (mm.) at a constant sample extension corresponding to a base length of 2 cm. along the alveogram.

Structural relaxation curves were constructed in which the rest period was plotted against resistance for the various sets of conditions mentioned. The data were selected in such a manner as to show the structural relaxation process in bromated and unbromated doughs for a wide variety of conditions. Figure 19 shows the structural relaxation curves for 0, 1, 2 and 4 hr. reaction times and 0, 10 and 30 p.p.m. potassium bromate.

These plots show that for 0 p.p.m. potassium bromate there is very little differentiation between the 0, 2 and 4 hr. reaction time curves.

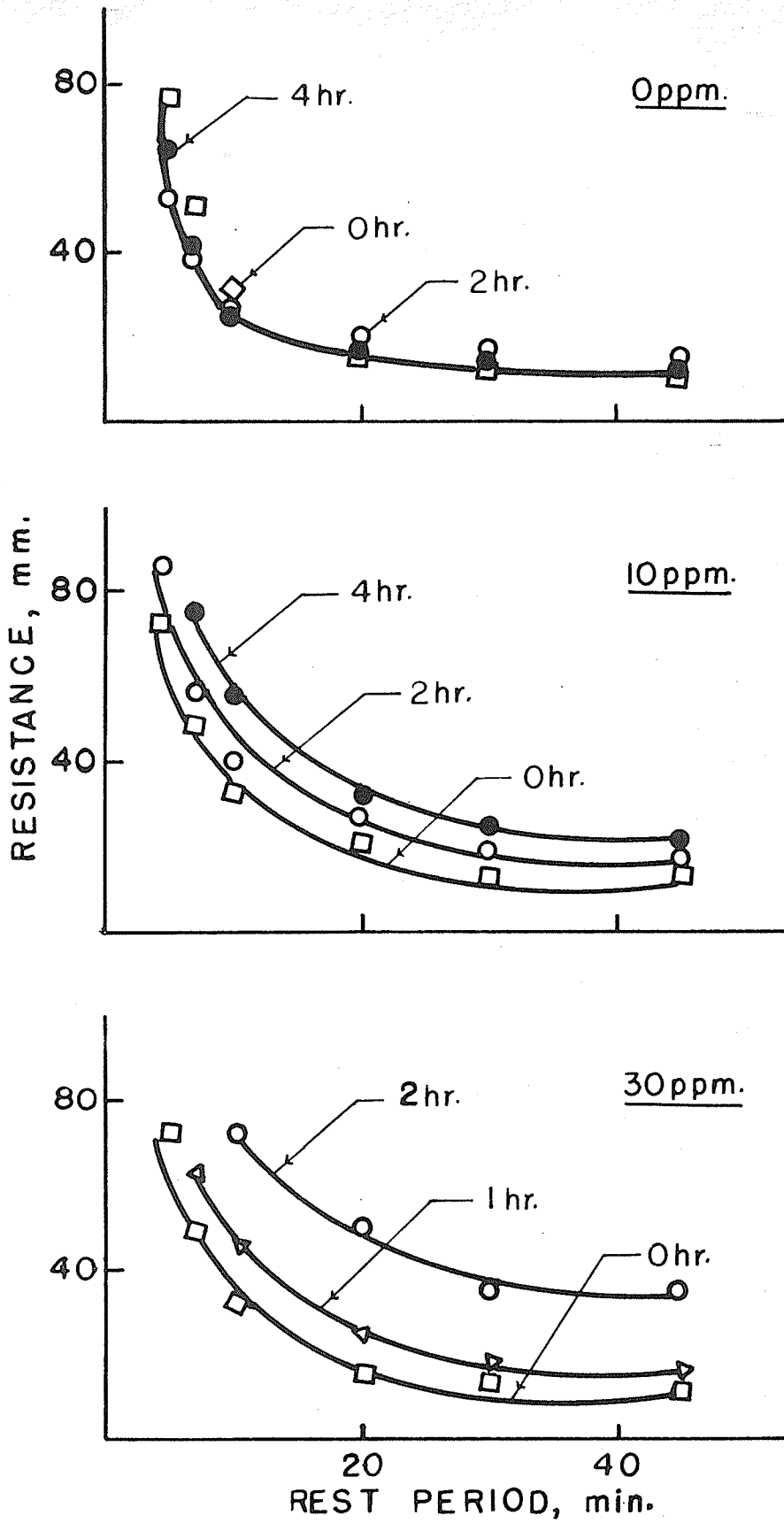


Fig. 19

Structural relaxation curves for 0, 1, 2, and 4 hr. reaction times and 0, 10 and 30 p.p.m. potassium bromate.

However for doughs treated with 10 and 30 p.p.m. potassium bromate it is found that doughs increase in their resistance to extension as the reaction time and bromate concentration increase.

More quantitative information about the effect of potassium bromate dough may be obtained by the characterization of relaxation curves as suggested by Dempster et al (10) in the analysis of extensograms.

The equation of an equilateral hyperbola referred to its asymptotes as axes can be used to characterize structural relaxation curves. The equation of an equilateral hyperbola, referred to its asymptotes as axes, and having as asymptotes the line $t = 0$ and $R - R_A = 0$ is

$$t(R - R_A) - C = 0 \quad (1)$$

A more useful form of this equation is:

$$Rt = R_A t + C \quad (2)$$

where R = resistance to extension of a dough at a rest period t .

R_A = the asymptotic resistance (the resistance approached at infinite rest period)

C = the relaxation constant

Two constants are thus necessary to define relaxation curves, namely the asymptotic resistance R_A and the relaxation constant C . Equation (2) shows that a plot of resistance x rest period vs. resistance is linear. The intercept at the axis gives the relaxation constant C and the slope gives the asymptotic resistance R_A .

The experimental data were therefore plotted as the product of resistance x rest period vs. rest period to obtain the rate constant C and the slope gives the asymptotic resistance R_A .

Figure 20 shows a set of plots of resistance x rest period vs. rest period for 0, 10 and 30 p.p.m. potassium bromate. Regression lines were calculated for these data and are shown as solid lines on the plots. These lines are extrapolated (dashed lines) to the ordinate axes. The intercept giving the relaxation constant C was calculated from the regression equations.

The progressive changes taking place in the dough as a result of the bromate reaction can be followed by plotting the relaxation constants against reaction times as shown in Fig. 21 where relaxation constants are plotted for reaction times of 0, 1, 2, 3 and 4 hr. and 0, 10, and 30 p.p.m. potassium bromate.

The relaxation constant gives a measure of the time taken for a dough to "relax" such that a dough giving a high relaxation constant is very slow to relax whereas if a low relaxation constant is recorded the dough relaxes rapidly. Figure 21 shows that the rate of relaxation for the 0 p.p.m. potassium bromate curve is quite rapid and increases with increase in reaction time as illustrated by the negative slope. For 10 and 30 p.p.m. potassium bromate the relaxation rate is progressively slower and the time taken for the dough to "relax" increases as the reaction time increases.

The most significant conclusion that can be drawn from this bromate study with the alveograph is the fact that the alveogram analysis, which includes the construction of relaxation curves and their hyperbolic characterization, can be treated in a strictly analogous manner to the

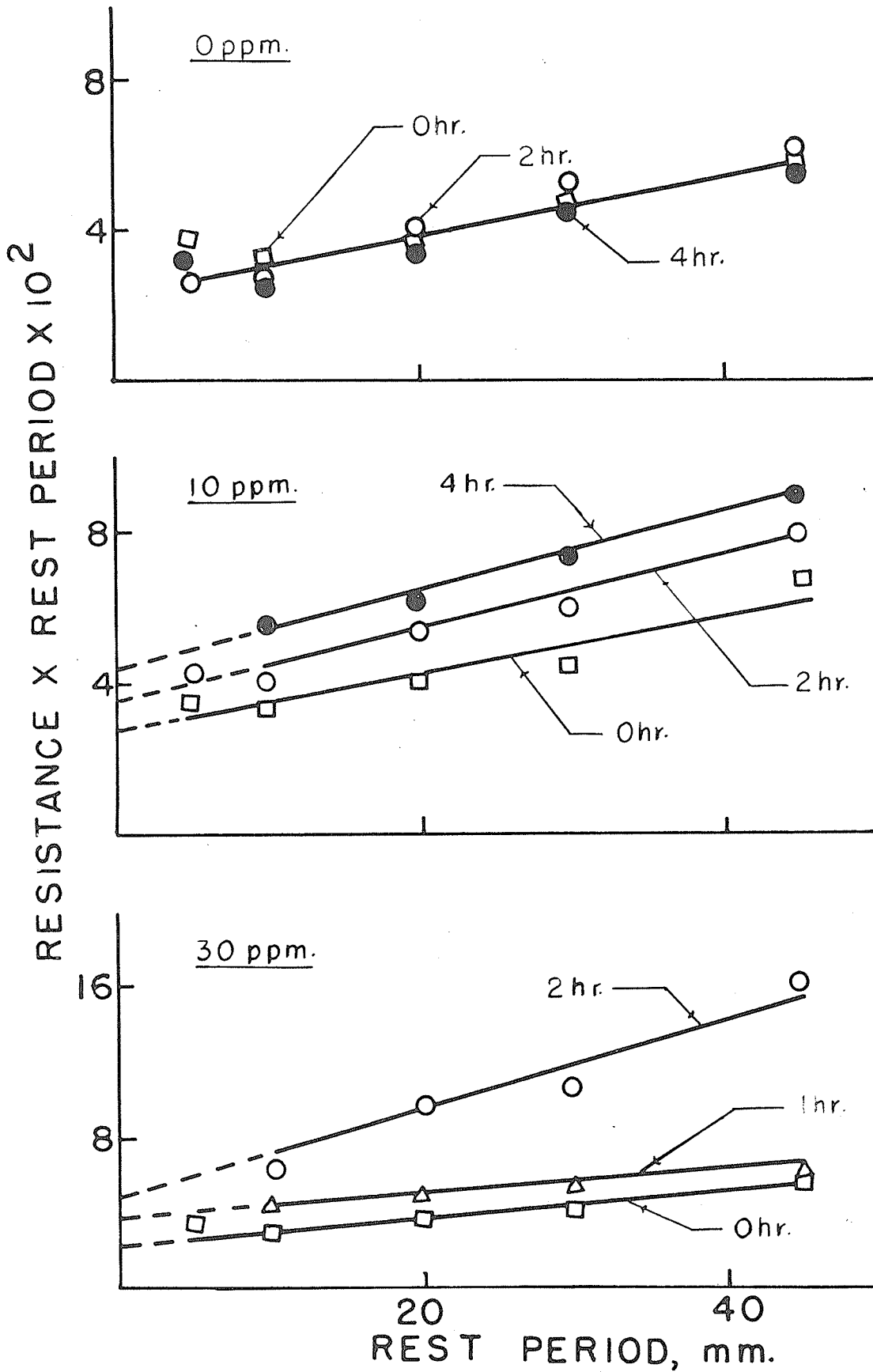


Fig. 20

Graphs testing the validity of the hyperbolic characterization of structural relaxation curves obtained from alveograms for bromated and unbromated doughs.

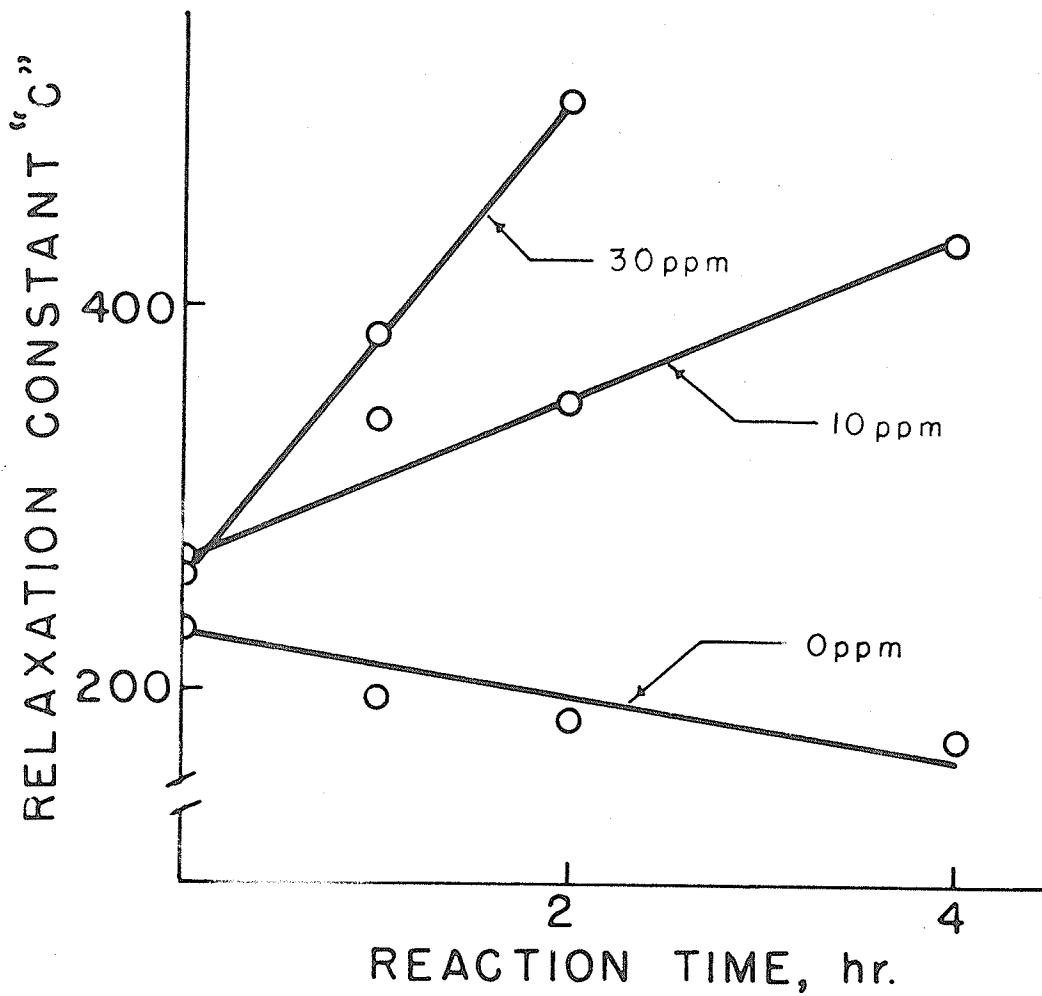


Fig. 21

The effect of increased reaction time and bromate concentration on the relaxation constant "C".

analysis of extensograms proposed by Dempster et al. A further study would have to be made to determine the advantages or disadvantages of the one instrument over the other.

Significance of Other Alveogram Constants

Variation of Dough Membrane Thickness at Alveogram Maximum. Perhaps the most important standard measurement made with the alveograph is the maximum height on an alveogram. This measurement, described by Chopin as indicating the tensile strength of a dough, is used extensively in routine dough testing with the alveograph for the determination of breadmaking quality of flour.

Because of the importance which has been placed on the alveogram maximum, an analysis of data was carried out to determine the variation of thickness of dough bubbles at the alveogram maximum.

The variation of dough membrane thickness at the alveogram maximum was examined for sets of alveograms obtained from dough samples subjected to a wide variety of conditions including different bromate concentrations, reaction times, rest periods and flow times. Tables VI, VII and VIII illustrate the variation in dough membrane thickness of dough bubbles at alveogram maximum for bromate concentrations of 0, 10 and 30 p.p.m. KBrO_3 , reaction times of 0, 1, 2, and 4 hr., rest periods of 5, 7, 10, 20, 30, 45 min. and flow times of 25.1 ml./sec., 19.0 ml./sec. and 14.8 ml./sec. Column (1) in each table gives the rest period for individual alveograms and columns (2) to (5) give reaction times for sets of alveograms.

Table VI

Variation of Dough Membrane Thickness at Alveogram Maximum
with a Flow Time of 25.1 ml./sec.

0 p.p.m. KBrO_3

Rest Period	0 Hr.	1 Hr.	2 Hr.	4 Hr.
min.	mm.	mm.	mm.	mm.
5	0.68	0.81	0.81	0.81
7	0.81	0.81	0.81	0.81
10	1.04	1.04	0.81	0.81
20	1.04	1.04	0.85	1.04
30	1.04	1.04	0.85	1.04
45	1.04	1.04	1.04	1.04

10 p.p.m. KBrO_3

5	0.81	0.81	0.68	-
7	0.81	0.81	0.81	0.81
10	0.81	0.81	0.81	0.81
20	0.84	1.04	0.84	0.81
30	1.04	1.04	1.04	0.92
45	1.04	1.04	1.04	0.92

30 p.p.m. KBrO_3

5	0.68	-	-	-
7	0.68	0.81	-	-
10	0.92	0.81	0.68	-
20	0.92	0.92	0.68	-
30	0.92	1.04	0.80	-
45	0.92	1.04	0.80	-

Table VII

Variation of Dough Membrane Thickness at Alveogram Maximum
with a Flow Time of 19.0 ml./sec.

0 p.p.m. KBrO_3

Rest Period	0 Hr.	1 Hr.	2 Hr.	4 Hr.
min.	mm.	mm.	mm.	mm.
5	0.81	0.81	0.70	0.81
7	0.81	0.81	0.70	0.81
10	0.99	0.81	0.99	0.99
20	1.06	0.99	1.32	1.32
30	1.06	1.32	1.32	1.32
45	1.06	1.32	1.32	1.32

10 p.p.m. KBrO_3

5	0.99	0.81	0.70	-
7	0.99	0.81	0.81	-
10	0.99	0.81	0.81	0.81
20	0.99	0.99	0.99	0.81
30	1.32	1.32	1.32	1.32
45	1.32	1.32	1.32	1.32

30 p.p.m. KBrO_3

5	0.86	-	-	-
7	0.86	0.70	-	-
10	0.86	0.70	0.70	-
20	0.92	0.81	0.70	-
30	0.92	0.99	0.99	0.70
45	0.92	0.99	0.99	0.70

Table VIII

Variation of Dough Membrane Thickness at Alveograph Maximum
with a Flow Time of 14.8 ml./sec.

0 p.p.m. KBrO_3

Rest Period	0 Hr.	1 Hr.	2 Hr.	4 Hr.
min.	mm.	mm.	mm.	mm.
5	0.70	0.72	0.61	0.72
7	0.70	0.72	0.61	0.72
10	0.81	0.81	0.81	0.81
20	0.93	1.10	0.93	1.02
30	0.93	1.10	0.93	1.02
45	0.93	1.10	0.93	1.02

10 p.p.m. KBrO_3

5	0.61	0.76	0.61	0.61
7	0.61	0.76	0.61	0.61
10	0.61	0.76	0.74	0.61
20	0.93	1.10	0.93	0.81
30	0.93	1.10	0.93	1.10
45	0.93	1.10	1.10	1.10

30 p.p.m. KBrO_3

5	0.75	-	-	-
7	0.75	0.61	-	-
10	0.75	0.61	0.76	-
20	0.93	0.81	0.76	-
30	0.93	0.81	0.76	0.61
45	0.93	0.93	0.93	0.61

These results show that the maximum variations in membrane thickness are: 0.68 to 1.04 mm. (Table VI), 0.70 to 1.32 mm. (Table VII), and 0.61 to 1.04 mm. (Table VIII). A significant conclusion that may be drawn from these results is that there is a large variation in the membrane thickness at the alveogram maximum for alveograms selected for a wide variety of conditions. Consequently alveogram maxima cannot be used as a basis of measurement for relaxation curves.

The data also show that the variations in membrane thickness increase as the flow time increases. It is noted that the pressure variations, resulting from choosing the alveogram maximum instead of a constant membrane thickness, are quite large.

The Significance of the Square Root of the Volume and the Extensibility

The square root of the volume and the extensibility of an alveogram have been used by Chopin and other investigators to determine flour quality. These two standard measurements are related to one another in that as the volume of a dough bubble increases the extensibility of an alveogram increases. Early investigators wrongly assumed that the square root of the volume was also proportional to the surface area of the bubble. From first principles it can be shown that a more complex function exists between these two factors. The square root of the volume is represented by a $3/2$ power of the radius and the surface area of a bubble by a second power.

The extensibility and the square root of the volume were examined

for a wide selection of alveogram data to determine whether these measurements could be applied to the study of the structural relaxation of bromated and unbromated doughs.

The results obtained for the extensibility and square root of the volume in the relaxation study differ widely and thus it is necessary to discuss these two factors separately.

Plots were made of the square root of the volume vs. resistance for the conditions 0, 4 hr. reaction time 0 p.p.m. potassium bromate, 4 hr. reaction time 10 p.p.m. potassium bromate and 2 hr. reaction time 30 p.p.m. potassium bromate as shown in Fig. 22. The majority of these plots show a general relationship indicating that the square root of the volume may be a function of the resistance. This assumption is further substantiated by the rest period x square root of the volume vs. rest period structural relaxation plots (Fig. 23) for the same conditions for rest periods of 5, 7, 10, 20, 30 and 45 min. These plots show a very good linear relationship. However, it is very doubtful whether the square root of the volume can be applied to structural relaxation studies because there is little or no differentiation between the curves for a wide variation in reaction times and bromate concentrations.

The variation of bromate concentrations and reaction times thus appears to have no effect on the square root of the volume measurement.

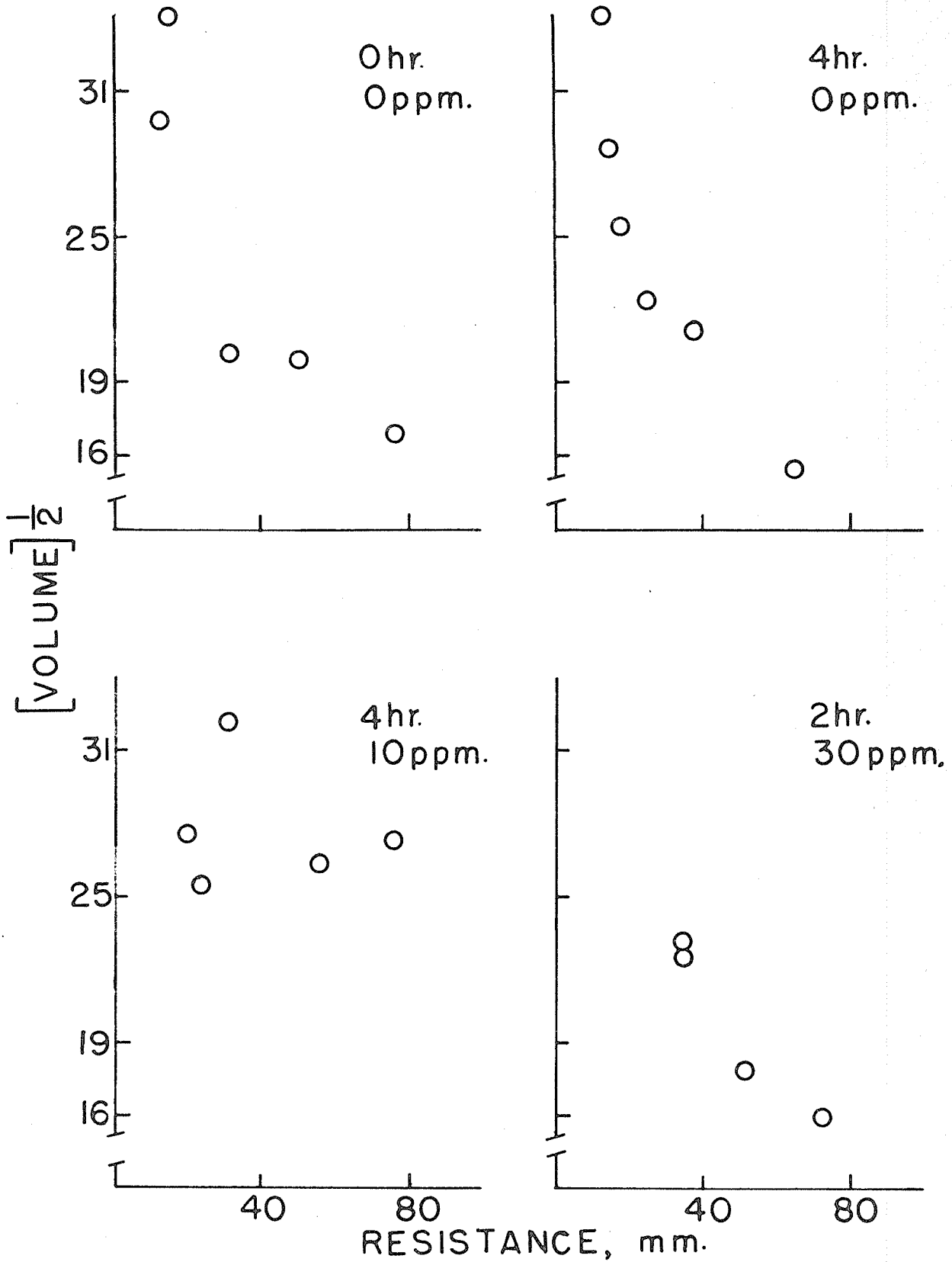


Fig. 22

Relationship between square root of the volume of a dough bubble and resistance for a variety of reaction times and bromate concentrations.

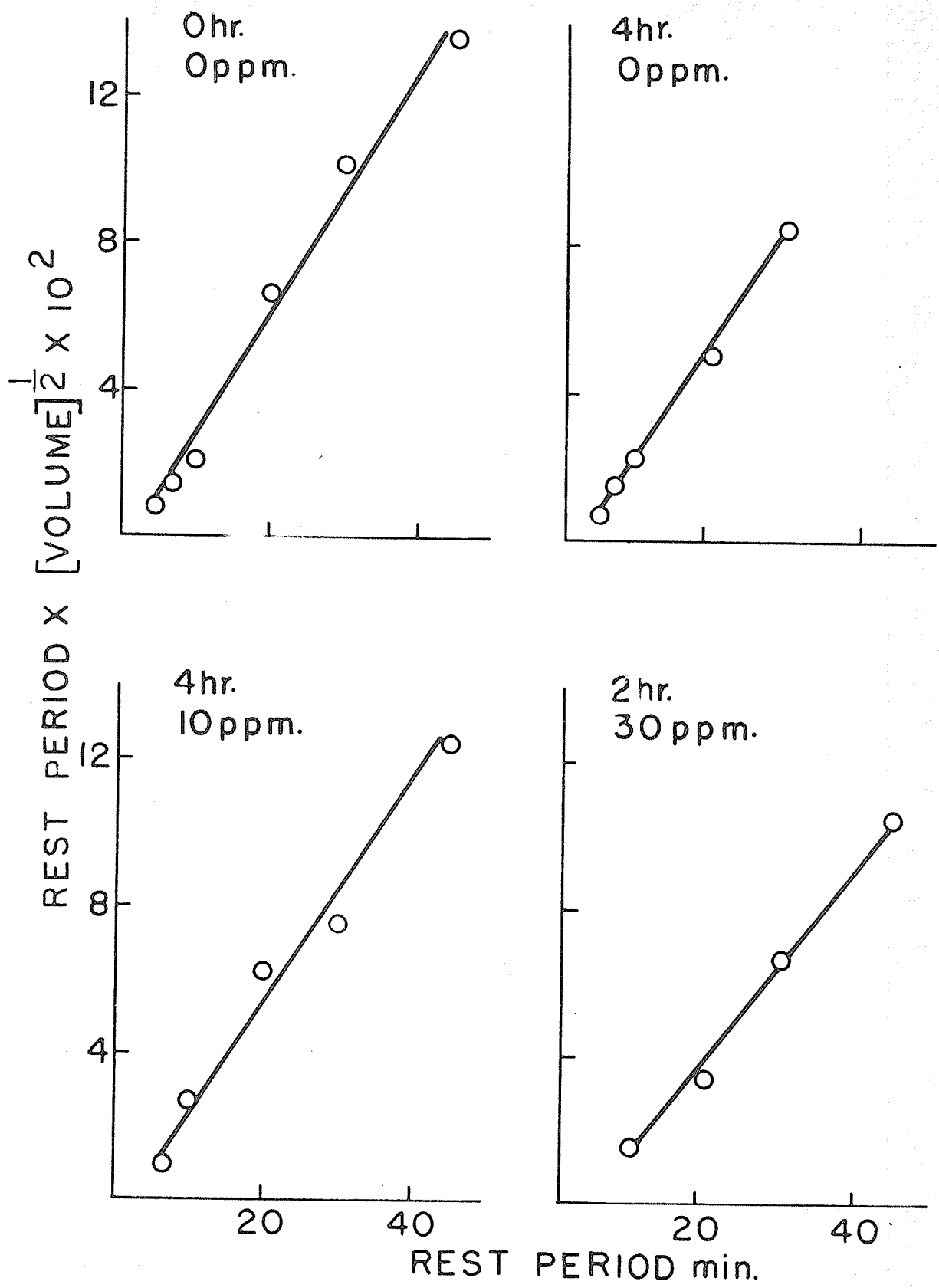


Fig. 23

Plot of rest period time x square root of volume vs. rest period for bromated and unbromated doughs.

Plots (Fig. 24) were also made of extensibility vs. resistance for the conditions 0, 4 hr. reaction time 0 p.p.m. potassium bromate, 4 hr. reaction time 10 p.p.m. potassium bromate and 2 hr. reaction time 30 p.p.m. potassium bromate. These plots indicate that a general relationship may exist between the extensibility and the resistance. This relationship is supported by the structural relaxation plots of extensibility x rest period vs. extensibility for the following conditions: 0, 2, 4 hr. reaction time 0 and 10 p.p.m. potassium bromate, and 0, 2 hr. reaction 30 p.p.m. potassium bromate. These plots (Fig. 25) show a very good linear relationship and what is more significant there is a steady downward displacement of the curves which increases for increasing reaction times and bromate concentrations. The extensibility is therefore found to decrease with increasing reaction time and bromate concentration.

A summary of these results indicates that the square root of the volume cannot be applied to structural relaxation studies. On the other hand the extensibility is found to be a fundamental measurement which can be used in the study of the structural relaxation of bromated and unbromated doughs.

The Significance of the Area Under an Alveogram Curve and the Work Function. Two standard measurements which have been used in the evaluation of flour quality are the area under an alveogram

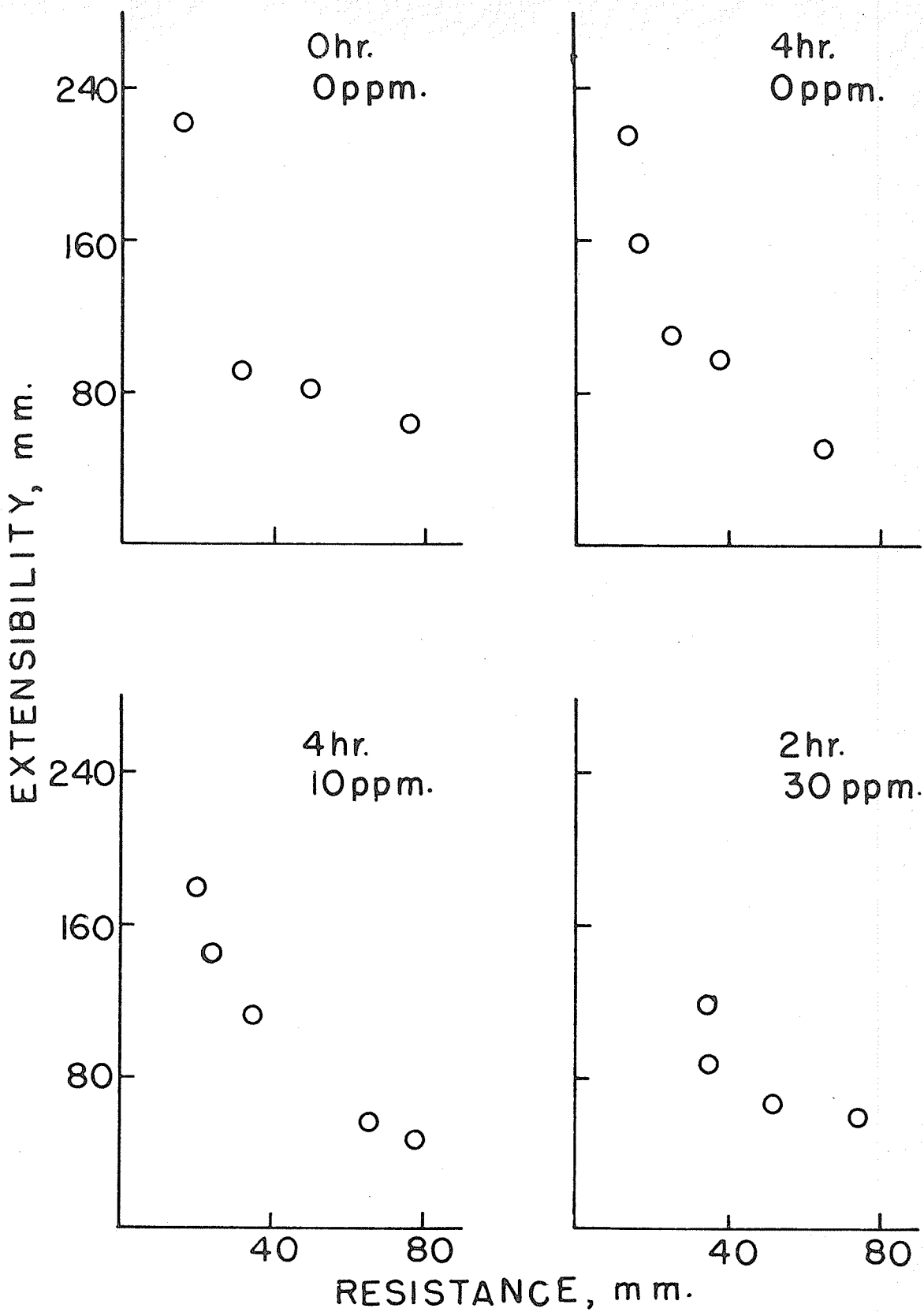


Fig. 24

Relationship between extensibility and resistance for a variety of reaction times and bromate concentrations.

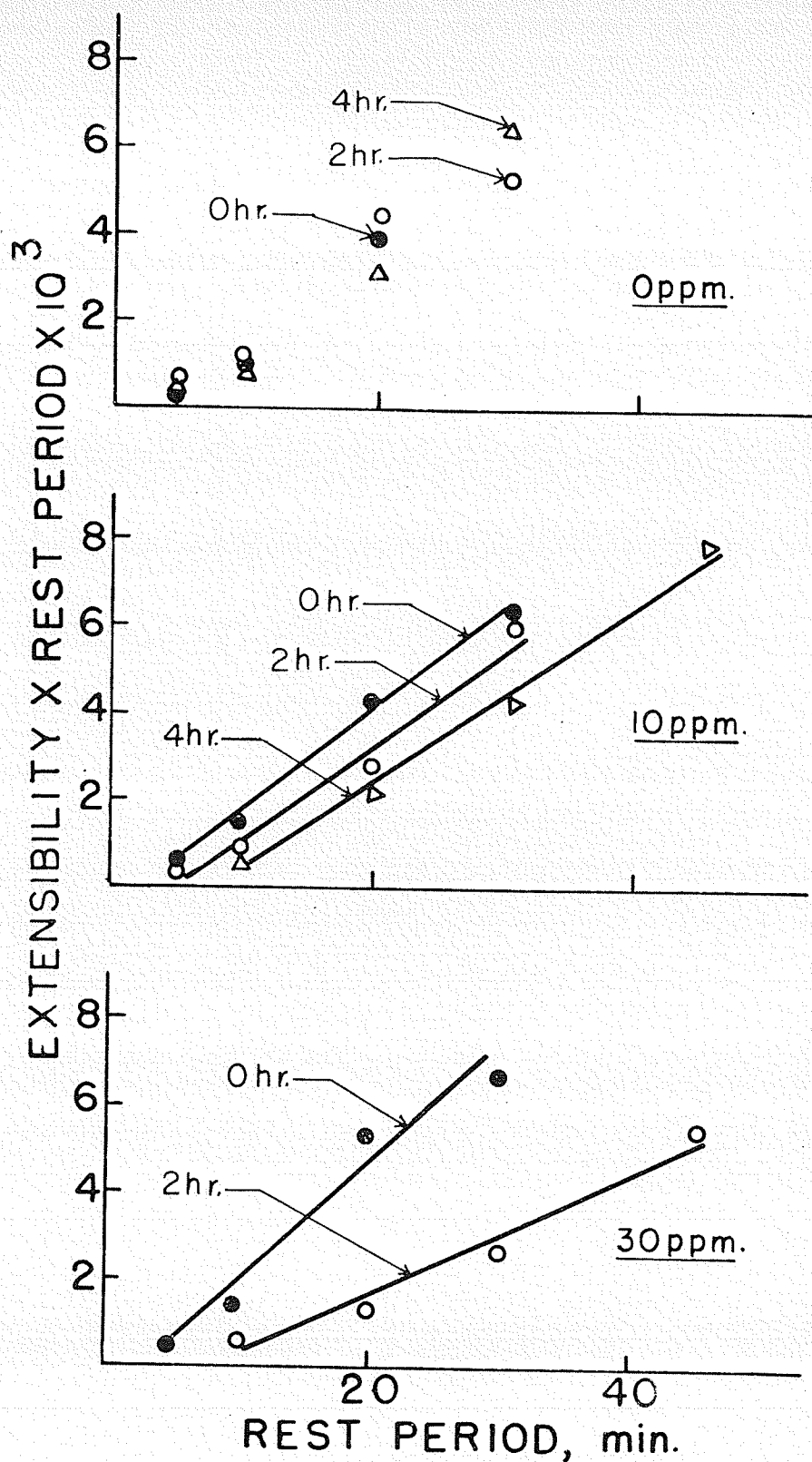


Fig. 25

Plot of rest period x extensibility vs. rest period for bromated and unbromated doughs.

curve and the work function. The area under the alveogram curve (measured with the aid of a planimeter) and the work function are related through the equation used for the calculation of the work function where:

$$W = \frac{K \times C \times S}{L}$$

where W = work function

C = manometer constant

S = area under the curve

L = length of curve.

These two measurements were examined for a wide selection of alveogram data to determine if they have any practical application in the study of structural relaxation of bromated and unbromated doughs at a constant sample deformation. The graphs constructed are not presented in this discussion because they do not serve to illustrate any definite trends or relationships between the factors involved. However the results obtained will be briefly summarized.

Plots of resistance vs. area under the alveogram curve and also the work function were made for the following conditions 0, 2, 4 hr. reaction time, 0 and 10 p.p.m. potassium bromate, 0, 1, 2 hr. reaction time and 30 p.p.m. potassium bromate. The random scatter of points on the graphs indicated no direct relationship between the resistance and the area under the alveogram curve or the work function. Furthermore, there appeared to be no definite trend between the curves for wide variations in reaction times and bromate concentrations. The structural relaxation plots of rest period x area under curve vs.

rest period and rest period x work function vs. rest period substantiated these findings. In the case of the area and the work function it was noted that only a very approximate straight line relationship existed and no definite trend between the curves for various reaction times and bromate concentrations was found.

It may therefore be concluded that the work function and the area under the alveogram curve cannot be applied to the study of structural relaxation at a constant sample deformation. No fundamental significance of these measurements could be established.

GENERAL DISCUSSION

Before the present study was undertaken the alveograph was used primarily to evaluate flour quality. This evaluation was carried out by a comparison of certain standard measurements made on alveograms obtained from different types of flour. These measurements included the maximum height of an alveogram, the area under the curve and the extensibility.

The present investigation constituted the first systematic attempt to derive data from the results obtained with this empirically-designed instrument, which can be used quantitatively to yield information about the structural relaxation process which takes place in bromated and unbromated doughs.

A major part of this thesis was devoted to showing that the alveograph is a suitable instrument for the study of structural changes occurring in dough. An examination of the geometrical considerations (volume, area and thickness of dough bubbles) involved in the operation of the alveograph as well as the pressure measurement recorded by this instrument revealed that structural relaxation curves could be constructed for sets of alveograms measured at a constant sample deformation. These curves give a quantitative measure of the time dependent changes taking place in bromated and unbromated doughs or in other words they relate structural changes taking place in dough.

Certain standard measurements including the alveogram maximum, area under the curve, extensibility, the work function and the square root of the volume of a dough bubble were tested to see if they were fundamental measurements which might be used in the construction of relaxation curves. The extensibility was found to be the only other measurement beside the resistance which might be used in the construction of relaxation

curves. It was found during the course of the investigation that the area of a dough bubble is a fundamental measurement. The volume being directly related to the area can also be considered as a fundamental measurement. However, it has been shown that the resistance is a more reproducible measurement than the volume or extensibility and therefore the resistance measurement is preferred for the construction of relaxation curves.

Because the alveograph inflates a dough into a surface i.e. a two dimensional deformation which is highly imitative of the formation of small bubbles in the structure of a fermenting dough, it may be as well, or better suited for assessing the physical properties that take place in dough as the Brabender extensograph.

Now that a method for using the alveograph to show the physical changes in dough has been developed further research may be carried on with this instrument to examine the elastoviscous properties of dough under a wide variety of conditions.

CONCLUSIONS

The object of this investigation was to develop a method of studying structural relaxation in dough using the Chopin alveograph as the dough testing instrument. An account of the main conclusions obtained from the investigation will now be given.

A method, analogous to that used for extensograph studies, was introduced for the study of structural relaxation of dough using the alveograph. This method provided for a reaction time, structural activation and a rest period.

In order that alveogram curves may be directly comparable to one another, a constant sample deformation was introduced for alveogram analysis. The measurement used for the deformation was the pressure recorded referred to unit dough membrane thickness. The linearity of the plot of resistance x area vs. area tested under a wide variety of conditions showed that pressure is directly related to dough membrane thickness. This plot also revealed that a set of alveograms belong to one family of curves.

Structural relaxation curves, for a wide variety of conditions, can be constructed from alveograms by plotting the resistance (recorded as height of alveogram curve in mm.) at a constant sample deformation vs. rest period. These relaxation curves can be further characterized by the equation of an equilateral hyperbola referred to its asymptotes as axes.

The extensibility was found to be a fundamental measurement which can be used in structural relaxation studies.

Other commonly used measurements including the alveogram maximum, square root of the volume, area under the curve and the work function were tested under a wide variety of conditions. No fundamental significance was found for these measurements and furthermore it was found that they could not be applied to structural relaxation studies.

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