

Water Self-Diffusion Coefficients and Dielectric Properties Determined for Starch-Gluten-Water Mixtures Heated by Microwave and by Conventional Methods¹

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ABSTRACT

Cereal Chem. 69(6):637-642

The effect of microwave and conventional heating on water mobility (self-diffusion coefficient, D) and dielectric properties was investigated. Wheat starch and vital wheat gluten were mixed in various proportions (100:0, 80:20, 50:50, 20:80, and 0:100) at three different moisture contents (54, 100, and 186%). Measurements were made for the individual powders and for each of the 15 combinations of starch, vital wheat gluten, and water before heating, after conventional heating, and after microwave heating. For the dry powders, no differences were found in dielectric properties, but differences were found in the mobility by pulsed gradient

spin-echo nuclear magnetic resonance. For nearly all of the mixtures, two D values were obtained at all three moisture contents. Moisture content had a significant effect on D values, as did heating. The difference between conventionally heated and microwave-heated samples was not great. The dielectric properties were affected by heating method—the dielectric constant was more dependent on moisture content than was dielectric loss, which showed very little variation over the three moisture contents.

The location and behavior of water during dough preparation and bread baking is not fully understood. Webb et al (1970) assessed and summarized what happens during this process. Fickian diffusion between starch and gluten, before and after baking, in a model system was determined by measuring moisture content (Willhoft 1971). The author suggested that there was more water associated with the gluten than with the starch in the dough and that the water moves from the gluten to the starch during conventional baking. Webb et al (1970) showed that the amount of water added to a dough has little effect on the gluten development or structure from 48 to 66% moisture (dry basis). Starch and gluten were examined by Wynne-Jones and Blanshard (1986) via nuclear magnetic resonance (NMR) transverse relaxation measurements, and they found that moisture content is very important for the mobility of the water.

Microwave oven use has increased in the last 20 years. This method of heating, an interaction of electromagnetic waves with water molecules (and other constituents), does not produce the same product as does conduction heating. Even if both methods of baking reach the same final temperature, the rate of temperature increase is usually much slower for conventional heating than it is for microwave heating, because of the high power levels and smaller product sizes associated with the latter method. In a cereal-based system, water, starch, and gluten could interact in different ways by the two modes of heating and result in different products. The effect of heating on molecular interactions requires more investigation.

Pulsed gradient spin-echo nuclear magnetic resonance provides a very specific method of examining water movement and the degree of water-polymer association without destroying the integrity of the sample. The use of a time-dependent (pulsed) field gradient to measure proton self-diffusion coefficients (D) was first proposed in 1965 for protons (Stejskal and Tanner 1965). The two field gradient pulses allow the nuclei to be positionally identified, and translational movement during the course of the experiment can be determined by the refocusing of the magnetization after the second radio frequency pulse (Blum 1986). The D values provide information on the Brownian (or random) motion of the nuclei of interest (Karger et al 1988). It should be emphasized that this method of tracing the movement of molecules leads

to a measurement of Brownian motion in the absence of concentration gradients, thus leading to a noninvasive determination of the true self-diffusion coefficient. As the water interacts with other substances present in the sample, all motion becomes slowed and diffusion decreases from that of free water.

The ability to determine D values and their relevance in food systems to characterize molecular-level events has been well established. Cheese was examined by Callaghan et al (1983a) to determine the association of the water with the protein. Another example is wheat starch pastes, where there was a lack of correlation between rheological measurements and D values (Callaghan et al 1983b). A nonlinear relationship between moisture content and self-diffusion coefficients for water in potato starch (Lechert et al 1980) and wheat endosperm (Callaghan et al 1979) has been demonstrated.

Water is a critical component of a food with regard to heating by electromagnetic energy. The water molecule possesses an electric dipole moment and is the primary component of a food that interacts with electromagnetic radiation at 2,450 MHz. The dielectric constant (k'), dielectric loss (k''), and attenuation factor (α) are collectively called dielectric properties. The variables are used to give an indication of how a food system will store the electromagnetic radiation (k'), which is related to polarity, and how it will transform or dissipate the electric energy into heat (k''). The third property is a measure of how the waves will be affected when they go from the surrounding air into the food medium. The inverse of α is penetration depth, which provides information on how far a wave will penetrate into the sample before being reduced to 36% ($1/e$) of its original intensity (Mudgett 1986). Food systems have not been widely characterized by their dielectric properties.

A relationship between magnetic and dielectric relaxation was first described by Bloembergen et al (1948) and has been investigated since then. While nuclear spin relaxation and dielectric relaxation arise from molecular rotation, self-diffusion, as measured by pulsed gradient spin-echo nuclear magnetic resonance, is a measure of Brownian translational motion. Because of water's importance for heating by microwave energy, it is of interest to examine the results for both types of measurement of water in foods.

Water relaxation has been investigated by a number of scientists. Adsorption of water onto silica gels was examined by both nuclear and dielectric relaxation (Woessner and Zimmerman 1963), and the authors felt there should be a relationship between the two sets of data. The fact that the data did not show a simple relationship was attributed to the anisotropic motion of the molecules. In a summary of an investigation that examined sugar-water solutions by nuclear relaxation and dielectric magnetic relaxation (Suggett 1976), it was found that the two methods examined two different motional properties of the system (ring and side chain

¹Published as paper 19669 of the contribution series of the Minnesota Agricultural Experiment Station based on research conducted under projects 18-027 and 18-063.

Presented at the AACC 76th Annual Meeting, Seattle, WA, October 1991.

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orientation motion, respectively).

In this study, the association of water with starch and vital wheat gluten (VWG) macromolecules was evaluated. The effects of heating (by conventional and microwave methods) to a level to allow starch gelatinization and protein denaturation were investigated for five combinations of starch and gluten at three moisture contents. Self-diffusion coefficient and dielectric property data were obtained for each system.

MATERIALS AND METHODS

Sample Preparation

All moisture contents are given on a dry basis (i.e., grams of water per gram of solids multiplied by 100). VWG (Whet-pro 80, Ogilvie Mills, Minneapolis, MN) gave the following proximate analysis: 8.2% moisture, 76.5% protein, 0.15% fat, and 0.41% ash, and wheat starch (Aytex-P, General Mills Chemicals, Inc., Minneapolis, MN) gave the following proximate analysis: 12.1% moisture, 0.45% protein, 0.15% fat, and <0.02% ash. A $3 \times 3 \times 5$ factorial study was done. The treatment of the sample—unheated, microwave (MW) heated, and conventional (CV) heated—was one main effect. Another main effect was the moisture content (not including the water present in the powders) of the sample—54, 100, and 186%. The third main effect was the composition of the sample. Five ratios (100:0, 80:20, 50:50, 20:80, and 0:100) of starch and gluten were examined. In general, to prepare the samples, the dry powders were weighed and mixed, the appropriate amount of water was added, and the ingredients were thoroughly combined.

Specifically, for the unheated samples, a 2-g ball was made by weighing the water with the dry ingredient mixture. Immediately after mixing, D values were obtained. For the heated samples, an 80-g ball was made, and the water was measured volumetrically. The samples were heated in a 250-ml beaker to a final temperature of 95°C and were allowed to cool for 10–15 min before measurements were taken.

It was not possible to obtain data for some of the heated samples. At 54% moisture, a homogeneous mixture of the 80-g sample of 20:80 and 0:100 compositions was not possible. At 186% moisture, the 100:0 sample could be easily made up, but it was too sticky after heating to insert into the NMR tube for measuring.

Heating and Determination of Self-Diffusion Coefficients

Samples were heated in a household MW oven (700W Panasonic) equipped with a turntable at 700W for 45 sec or in a household CV oven at 190°C for 25 min.

Self-diffusion coefficients (D , m^2/sec) were determined at 30°C using a JEOL FX60 pulse Fourier transform spectrometer with modifications described elsewhere (Callaghan et al 1980) using the method of Stejskal and Tanner (1965). Four-millimeter (i.d.) NMR tubes were used for obtaining D values. For single-component diffusion, the ratio of the echo signal with and without the application of the pulse gradient (i.e., the echo attenuation) was written according to the Stejskal-Tanner relation (Stejskal and Tanner 1965) as follows:

$$E/E_0 = \exp(-kD) \quad (1)$$

where $k = \gamma^2 \delta^2 G^2 (\Delta - \delta/3)$, and γ is the nuclear gyromagnetic ratio, δ is the gradient pulse duration, G is the gradient amplitude, and Δ is the time separation of the two gradient pulses. In the experiments reported here, G was 4.72 T/m, Δ was 5 msec, and δ varied between 0.1 and 2 msec. Where two components are present, equation 1 may be modified to read

$$E/E_0 = A_n \exp(-kD_1) + (1 - A_n) \exp(-kD_2) \quad (2)$$

where A_n is the normalized amplitude of the component with diffusion coefficient D_1 , and $(1 - A_n)$ is the normalized amplitude of the component with diffusion coefficient D_2 . Fits using this two-component relation were carried out using a program based on a least squares method. The D values were obtained with a calibration accuracy of $\pm 0.5\%$.

Heating and Determination of Dielectric Properties

Samples were MW heated in a hybrid oven (Hung 1980) at 400W for 90 sec and CV heated in a household oven at 190°C for 25 min. A reflectance method was used with a Hewlett-Packard network analyzer (model 8753A) with a Hewlett-Packard dielectric probe (model 85070A). The values for k' and k'' were simultaneously obtained at 2,450 MHz at 22°C. The attenuation factor (α , cm^{-1}) was calculated with the following equation:

$$\alpha = \frac{2\pi}{\lambda} \sqrt{\frac{k' \sqrt{1 + \tan^2 \delta} - 1}{2}} \quad (3)$$

where λ is equal to 12.245 cm and $\tan^2 \delta$ is equal to the ratio of k'' to k' (von Hippel 1954).

Determination of Isotherms

Approximately 2 g of the VWG and starch powders (in triplicate) was placed in six desiccators, at 23°C, each containing a saturated salt solution to cover a range of water activities (indicated in parentheses): LiCl_2 (0.11), MgCl_2 (0.32), K_2CO_3 (0.43), $\text{Mg}(\text{NO}_3)_2$ (0.53), NaCl (0.75), and BaCl_2 (0.90). Toluene was put in the highest water activity desiccator to prevent mold growth. Samples were weighed until the weight change was minimal (less than 4 mg for two consecutive weighings), about three or four weeks. The isotherms were calculated by a computer program, which also determined the Brunauer-Emmett-Teller and Guggenheim-Anderson-de Boer monolayer values.

Statistical Analysis

The unbalanced data sets of D , k' , k'' , and α were each analyzed by an analysis of variance for main effects and all interactions using the Statistical Analysis System (SAS Institute 1987).

RESULTS AND DISCUSSION

Dry Powders

The VWG powder had a proton D of $2.6 \times 10^{-12} \text{ m}^2/\text{sec}$. This is about three orders of magnitude lower than water ($3 \times 10^{-9} \text{ m}^2/\text{sec}$), which indicates a very limited motion of the water molecules. Although the starch had a higher moisture content than did the VWG, a proton NMR signal could not be obtained, preventing a determination of a value of D . Provided that we neglect the effect of transverse relaxation, the magnitude of a signal should be proportional to the number of contributing nuclei. One simplistic explanation for the lack of a starch proton NMR signal might be to assert that the VWG contained many more ^1H nuclei. The percentage of protons, as a total of all atoms present in the sample on a molecular weight basis, was calculated. In the calculation of the protons in VWG, the percentage of each amino acid that was proton, the percentage of the total sample that each amino acid comprised (based on the amino acid profile obtained for the sample), and the protons on the starch (16.9%) were used. The starch macromolecular composition was taken to be repeating glucose units, and the entire sample was assumed to be starch and water. The results of these calculations were that 6.9% of VWG and 6.7% of starch was proton, which does not show a difference that would be great enough to cause the difference in the amount of signal.

A more likely hypothesis is that the water molecules in the "dry" starch were closely associated with the rigid polysaccharide structure and, therefore, their protons had a very short transverse relaxation time, making them "invisible" in the present experiment. It is certainly the case that the "degree of adsorption" of the water in the VWG and starch is approximately the same. From the isotherms (Fig. 1), the Brunauer-Emmett-Teller equations gave a monolayer value of 0.046 and 0.070 g of water per gram of solid for VWG and starch, respectively, which corresponds to water activity levels of about 0.08 and 0.10. These data agree with previous studies (Bushuk and Winkler 1957, Volman et al 1960, French 1984) and the Guggenheim-Anderson-de Boer monolayer (0.0483 and 0.0964 g of water per gram of solid for

VWG and starch, respectively). The monolayer values, on a dry basis, are not quite half the moisture contents for both the VWG (0.082) and starch (0.120), which means the water would be in about the same "layer" on the macromolecules. What is not known is the distribution of this water. Possibly, the water that interacts with the protein is located in specific regions or local areas where it would be quite mobile and give a signal. Bryant and Shirley (1980) found that water at a protein surface does exhibit some motion and is not rigidly held. For the carbohydrate, it is possible that the water is more evenly distributed and, thus, is too immobile to give a signal. According to French (1984), for starch the water will not be evenly distributed at the monolayer and not all the glucose molecules would have a water molecule (0.66 molecules of water per glucose unit). Volman et al (1960) thought this amount of water would be shared between the glucose molecules and fit well into the amylose helix, where it would likely be quite immobile.

The starch had a k' of 2.23, a k'' of 0.23, and an α of 0.0390, values that were comparable to the values for the gluten, which were 2.46, 0.13, and 0.0212, respectively. As expected, these values indicate that for both dry powder samples, there was very little interaction of the water with the electromagnetic radiation or dissipation of the electrical energy into heat energy. The small amount of mobility observed with the D value for the water in the gluten was not enough to allow movement of the dipole to any great extent.

Hydrated Samples

The statistical analysis indicated that the three main effects and all the interactions were significant ($P \leq 0.05$) for D and for the three dielectric properties.

For all of the mixtures, except for the 100:0, 54% moisture sample after heating, two values for self-diffusion were evident from the plot of $\log(\text{echo attenuation})$ vs. $\delta^2(\Delta - \delta/3)$ as shown in Figure 2. The signal was composed of a faster, more free population of water (the line with a more vertical slope) and a slower, more inhibited population (the line with a more horizontal slope). In all cases, the signals for the two populations appeared to have the same chemical shift, except for the 0:100, 54% moisture sample, when the two signals could be resolved. The NMR spectrum of this sample was examined with and without the field gradient on and then compared with a pure water peak. These results showed that the peak with the same chemical shift as the water was most attenuated when the field gradient was on, which indicates that the population of water most similar to pure water is attenuated most during the experiments. The other signal is a slower moving component that interacts more closely with the macromolecules so that the frequency of the NMR signal is shifted away from that of water and has a D value of about an order of magnitude (or less) slower than the fast component of that sample.

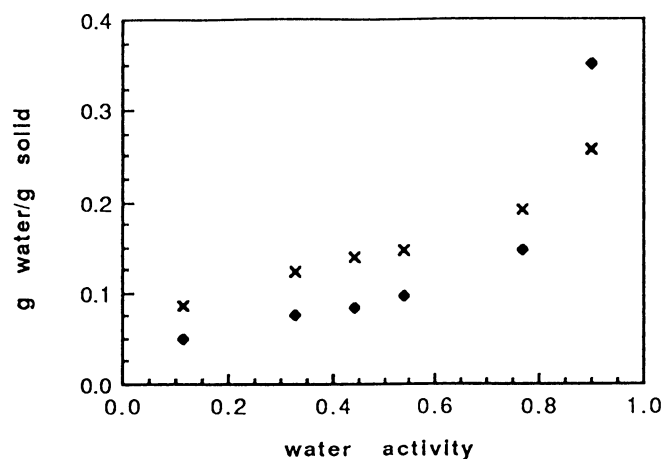


Fig. 1. Sorption isotherm for starch (x) and vital wheat gluten (◆) powders at 23°C.

An alteration of the length of time between the field gradient pulses resulted in no considerable change in the less-bound value and in different values of D for the more bound water, so the values for the more bound water cannot be well quantified. The reliability of the slow component value was minimal, but general comments can be made. For the unheated samples, the contribution of the slow component to the signal was between 2 and 25%. For each composition, the proportion of the slow component diminished with increasing moisture content. At the higher moisture contents, most of the signal comes from the more mobile water, and the amount of water interacting with the macromolecules is a very small portion of the total water in the sample. For the heated samples, the slow component contributed between 4 and 30% of the total signal. The moisture content did not have as great an effect on the proportion of the signal between the fast and slow component after heating as it did on the unheated samples. The 100 and 186% moisture content samples were quite similar in the proportion of the slow component, and the 80:20 CV-heated sample was the same for all three moisture levels.

In the above discussion, it must be remembered that these "fast" and "slow" components are relative terms and are for a mixed population. The Brownian motion of the slow diffusing group of water molecules was more inhibited by macromolecular interactions than was that of the fast diffusing group, with the latter being inhibited to some extent because the D values are less than that for free water. The number of "layers" of water that contribute to each component cannot be determined. The discussions of the NMR data that follow consider only the behavior of the faster, more mobile, component behavior.

The 100:0, 54% sample had only one population after heating. This could mean that all of the protons were in one environment producing one signal. Another explanation for the behavior is that the exchange between the populations is faster than the delay between the gradient pulses, thereby preventing both populations from being resolved.

Unheated Samples

The D value of the dough varied significantly ($P \leq 0.05$) with the amount of water (Fig. 3A); the more water that was present, the higher proportion of more mobile protons and the greater the diffusion coefficient measured. When any gluten was added to the starch, the value of D decreased, and the effect was more dramatic at the highest moisture content. On further addition of gluten, D was relatively constant. This suggests that the water-gluten interaction was "saturated" at low gluten contents and the subsequent D value of the water as gluten was increased was dominated by this interaction. Further evidence for this hypothesis is that the D value for water in the 100% starch sample at the highest moisture content was very close to the D value of free water. The proton mobility of the hydrated starch and VWG

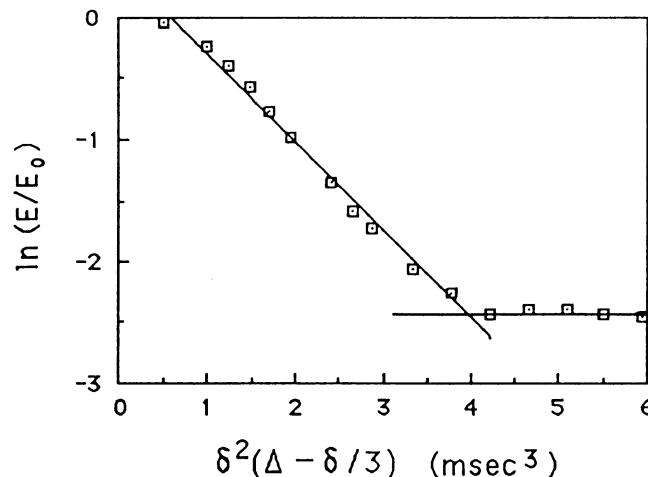


Fig. 2. Plot showing the effect of $(\delta^2[\Delta - \delta/3])$ increasing gradient pulse duration on $(\ln[E/E_0])$ echo attenuation for the pulsed gradient spin-echo nuclear magnetic resonance experiment.

was contrary to the mobility of the dry powders. That is, in the dry starch the water was very tightly held, and when more water was added, it did not interact with the macromolecules and remained quite mobile. In gluten dry powder, water had some mobility, and when more water was added, it interacted with the protein to result in a D value that was less than the D value of starch with the same amount of water. However, it is important to emphasize that in the dry powders, the mobility of the water, which is bound to the macromolecules, reflects the motion of the polymer rather than the degree of binding or interaction. In the case of the protein, it may well be that the water more strongly binds to the polymer but that the polymer is more rotationally mobile than is that of the starch. At higher water contents where the water is in exchange between "free" and "bound" states, the protein will, therefore, exert a greater attenuation of the translational diffusion of the water because of this stronger attraction. The dominance of gluten on the signal and the decrease in mobility in the presence of the macromolecules was determined by electron spin resonance (ESR) experiments using the probe TEMPO (2,2,6,6-tetramethylpiperidinyloxy) in water with starch (Pearce et al 1985) or gluten (Pearce et al 1988).

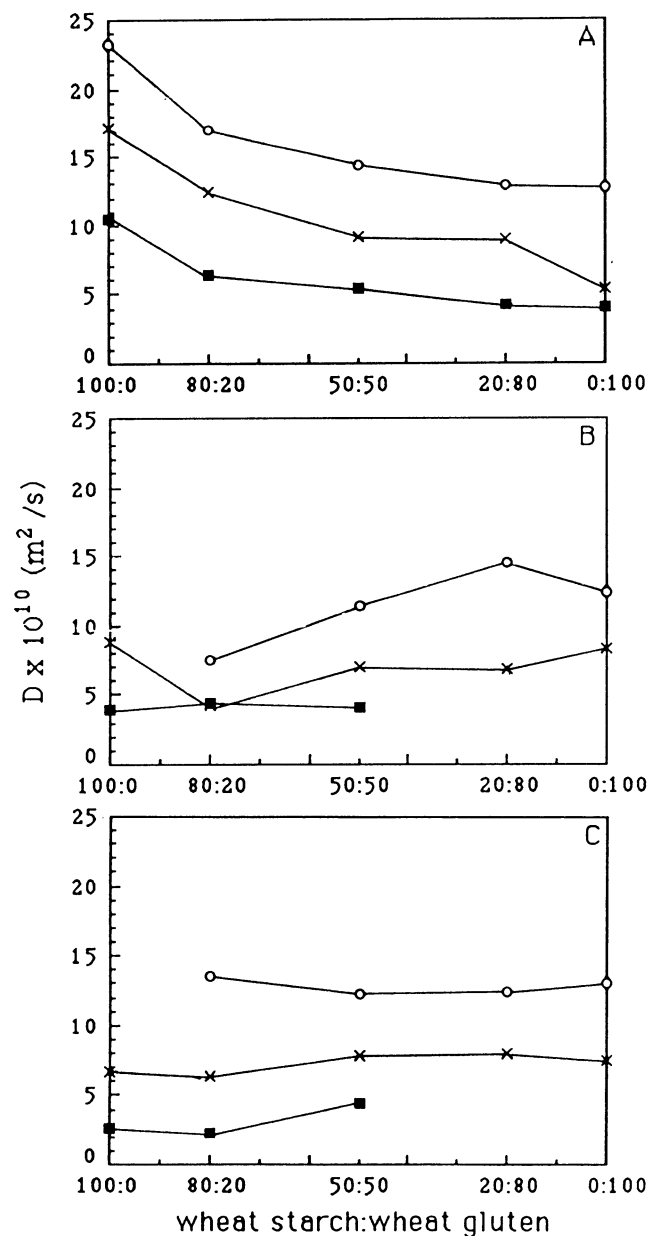


Fig. 3. Self-diffusion coefficients for unheated (A), conventionally heated (B), and microwave heated (C) starch and vital wheat gluten mixtures at three different moisture contents: 54% (■), 100% (x), and 186% (○) on a dry basis. (SD = 1.60×10^{-10} ; $P \leq 0.05$; $n = 6-24$).

Moisture content had a greater effect on the dielectric constant (Table I) than it did on the dielectric loss (Table II). As moisture content increased, the sample had increased ability to rotate and couple with the electromagnetic energy, but the k' values did not approach the k' value of water (76.92). As the amount of protein increased in the system, there was a decrease in k' , and the water appeared to interact more with the macromolecules, especially at the highest moisture content. For these compositions, the system appeared not to change over the three moisture contents; as more water was added, there was no change in the ability of the sample to transform the electrical energy to heat energy, as indicated by the k'' behavior, which is quite similar to that of water (9.38). The attenuation factor followed the same pattern as k'' (Fig. 4A), but the values were all greater than the value for water, which was 0.274.

Starch did not appear to absorb as much water as gluten because when equivalent amounts of starch or gluten were mixed with water at the higher moisture contents, the former was liquid and the latter was not. Thus, for the unheated mixtures, the physical appearance may provide an indication of the molecular behavior but not necessarily.

Heated Samples

Water loss during heating was primarily from the top surface because that was the only exposed surface area because the samples were heated in a beaker. The wide variety of compositions with this method of heating resulted in unusual physical appearances of the samples. The crumbly mixture of the high starch samples at 54% moisture became slightly more cohesive, although they remained somewhat crumbly after heating. At 100% moisture after MW heating, the center was an opaque gel, and the outside was a white powder; after CV heating, the sample had an opposite

TABLE I
Dielectric Constant^a (k') for Unheated and Heated Samples

Heating Method and Moisture Content	Starch/Gluten Ratio				
	100:0	80:20	50:50	20:80	0:100
Unheated					
54% db ^b	24.40	22.71	22.82	21.78	18.61
100% db	41.37	33.08	33.27	31.39	30.73
186% db	55.15	54.27	40.38	38.56	38.10
Conventional heated					
54% db	16.54	24.03	19.70	NA ^c	NA
100% db	29.43	20.52	32.85	31.66	21.06
186% db	41.55	29.09	35.22	31.78	28.84
Microwave heated					
54% db	17.66	5.77	8.76	NA	NA
100% db	29.15	22.30	1.01	2.14	20.41
186% db	49.13	43.12	36.61	27.70	24.30

^aStandard deviation: 5.45 ($P \leq 0.05$; $n = 5-16$).

^bDry basis.

^cNot available.

TABLE II
Dielectric Loss^a (k'') for Unheated and Heated Samples

Heating Method and Moisture Content	Starch/Gluten Ratio				
	100:0	80:20	50:50	20:80	0:100
Unheated					
54% db ^b	7.08	7.05	7.18	6.99	5.61
100% db	8.95	7.77	8.75	8.76	8.81
186% db	9.46	9.74	8.21	8.11	8.23
Conventional heated					
54% db	5.21	7.59	5.57	NA ^c	NA
100% db	10.09	6.42	8.20	7.53	5.03
186% db	11.48	7.84	8.34	5.88	6.05
Microwave heated					
54% db	6.49	1.65	2.71	NA	NA
100% db	7.55	5.84	0.24	0.18	5.36
186% db	9.95	9.32	7.85	6.22	5.48

^aStandard deviation: 1.47 ($P \leq 0.05$; $n = 5-16$).

^bDry basis.

^cNot available.

appearance. Measurements of the "gel" region were taken. This same appearance was found by other investigators (Goebel et al 1984, Zylema et al 1985, Wynne-Jones and Blanshard 1986) when starch and water were MW and CV heated. The 50:50, 54% moisture sample exhibited a layering of the dough. It was interesting that the MW-heated samples resulted in a single air cell with a surrounding shell of dough, and the CV-heated samples resulted in numerous air cells and the whole mass of dough expanded.

The decrease in D with heating (from the unheated samples) is evident in Figure 3B and C. The effect of gluten on the signal that was evident in the raw dough data was minimized during heating because of a redistribution of water from the gluten to the starch or because the free water became bound to the macromolecules. Possibly, the starch transformations during heating (gelatinization), which would destroy the rigid structure, allowed greater interaction between the starch macromolecules and the water. As expected, the denaturation of the gluten did not appreciably change water behavior as is evident by the 100% gluten samples (Fennema 1977). For most of the heated samples, mois-

ture content had a significant effect on these results, as it did on the unheated samples. The largest difference between the self-diffusion coefficients for the MW- and CV-heated samples was found in the 80:20, 186% moisture sample.

At 100% moisture for the all-starch sample, there was a big difference between the dough and the CV-heated sample. This is contrary to the ESR investigations reported by Pearce et al (1985) for starch (at about 200% moisture), where there was no difference in the motion of the spin probe (TEMPO) after heating. For the all-gluten sample at 100% moisture, there was a significant difference between the unheated and heated samples that was not evident when the moisture content was 186%. When gluten was examined by ESR at about 100% moisture, it was found that after heating, there was an increased partitioning of the probe into a hydrophobic region of the sample (Pearce et al 1988). The motion of water measured by a spin probe is indirect and the self-diffusion measurement is direct, which could explain the difference in the results between the two methods.

It has been reported, based on water adsorption studies, that in a dough more of the water is associated with the starch than with the protein (Bushuk and Hlynka 1964), thus there would be more hydrogen atoms associated with starch-interacting water than with protein-interacting water. This appears contrary to what was found in this study, where the gluten water appeared to dominate the signal. During heating, the water redistributes itself when the protein denatures and the starch gelatinizes absorbing water given up by the protein (Bushuk and Hlynka 1964). When the samples were heated, there was no composition effect indicating that there may be an even distribution of water between the macromolecules. The self-diffusion data reported here support this idea.

Heating generally caused a decrease in the k' (Table I) and k'' (Table II) relative to the unheated samples. A redistribution of the water may have occurred in the samples during CV heating, as all three moisture contents did not have very different k' and k'' values. The changes in the sample that occurred during CV heating changed the ability of the water to couple with the microwaves and transform the electrical energy to heat. MW heating did not cause the same type of changes in the system that CV heating did, and the moisture content had more of an effect on k' and k'' . Composition played a large role in the MW-heated samples, especially at 100% moisture. The water appeared to interact more strongly with the denatured gluten than with the gelatinized starch as the high protein samples had lower k' and k'' values than the high starch samples at 186% moisture.

The attenuation factor for the heated samples (Fig. 4B and C) at 186% for all samples and 54 and 100% at 100:0 were not too different from the unheated samples. Although not exactly the same, the pattern of the attenuation factor plots followed more closely to k' than to k'' . It is very clear that MW heating has a much different effect on the transformations in the sample that determine how the electromagnetic waves are attenuated. The interaction between composition and moisture content is clearly evident for the MW-heated samples.

CONCLUSIONS

The dry powder data indicated that self-diffusion coefficients provide an indication of water behavior that is different from that expected from dielectric property, moisture content, or water activity determinations. The effects of heating, which allowed macromolecular transformations, were evident by the decrease in D value, which indicated an increased binding and redistribution of the water between the starch and gluten, with the starch having a greater influence. Another interesting conclusion is that there was not much difference between the CV- and MW-heated samples in terms of their D values. Thus, the types of transitions that are occurring during the heating process that cause the differences in water binding are not affected by the rate of heating. For all five ratios of composition, as moisture content increased, there was generally an increase in D and k' , but k'' was not affected as much by moisture content. The k' and k'' values of the heated

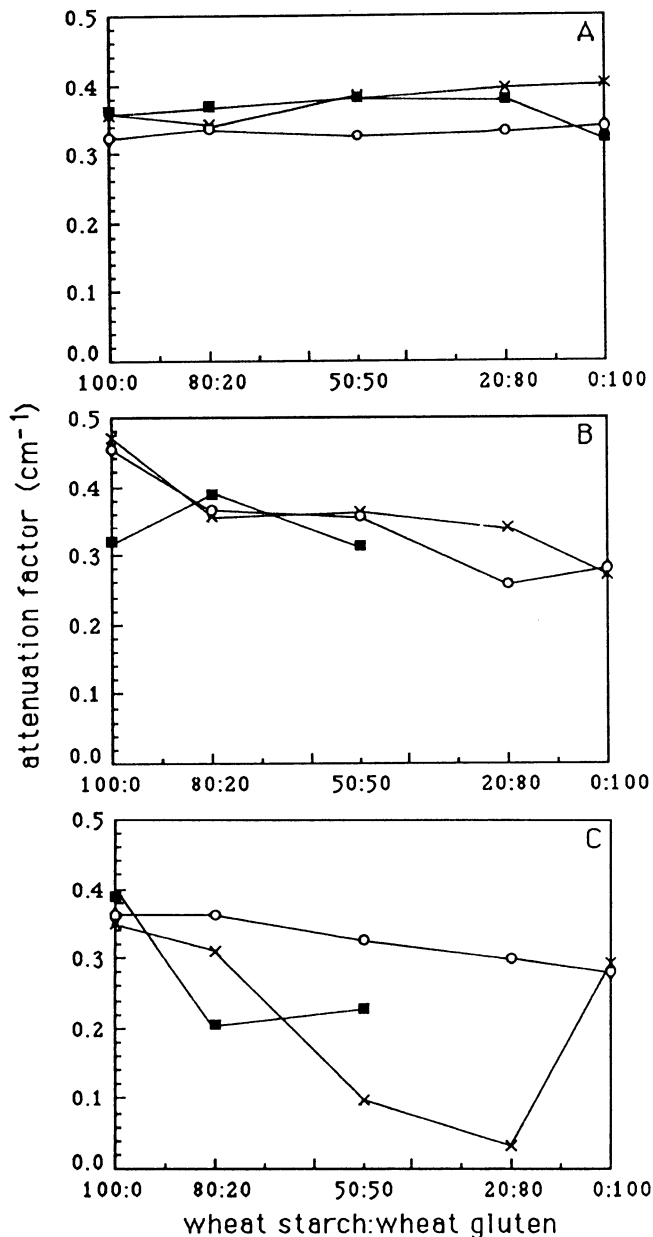


Fig. 4. Attenuation factors for unheated (A), conventionally heated (B), and microwave heated (C) starch and vital wheat gluten mixtures at three different moisture contents: 54% (■), 100% (x), and 186% (○) on a dry basis. (SD = 0.052; $P \leq 0.05$; $n = 5-16$).

samples were dependent on the ratio of starch to gluten, with the exact effect dependent on moisture content. The attenuation factor indicated that the two heating methods did cause different changes in the system in the way in which the sample interacted with the electromagnetic energy. Thus, this research has clearly demonstrated that more than one method is required to completely characterize the systems. And, although *D* and dielectric properties both measure water behavior, they examine different aspects of the microstructure of a starch-gluten-water system.

ACKNOWLEDGMENTS

We thank Haarmann and Reimer for their financial support of this research project. We also thank Andrew Coy and Kathy Nelson, who wrote the programs to calculate the self-diffusion coefficients and isotherms, respectively.

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[Received January 10, 1992. Accepted April 13, 1992.]