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FIBER COMPONENTS - QUANTITATION AND RELATIONSHIP TO CAKE QUALITY

Ву

Melissa Jeltema

AN ABSTRACT OF A DISSERTATION

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Abstract

Fiber Components - Quantitation and Relationship to Cake Quality

Layer cakes were prepared with 30% of the flour substituted with various cereal brans and cellulose products. The cereal brans included varieties of hard red, soft red, and soft white wheat brans as well as a commercial wheat bran, corn, soy, and oat bran. The cellulose products included mechanically ground cellulose, chemically hydrolyzed celluloses and prototypes coated with carboxy-methyl cellulose (CMC) and pectin. Parameters measured included cake volume, color, tenderness and batter viscosity. Sensory evaluation was conducted by a trained taste panel.

All of the brans and most of the cellulose types increased batter viscosity over the control. Most of the cellulose products had no effect on volume, tenderness, or most sensory parameters. Pectin coated cellulose decreased volume and sensory scores. As the percentage of pectin increased, the cakes showed further deterioration

in volume and sensory scores. Wheat brans had no significant effect on volume or most sensory parameters, while cakes containing other cereal brans differed significantly from the control and cakes containing wheat brans. The cakes made from non-wheat brans were less tender and more moist when compared to cakes containing wheat bran. The cakes containing non-wheat bran showed decreased sensory scores when compared with the control or cakes containing wheat bran. The cakes containing non-wheat bran also varied from each other in objective and sensory scores. Each bran increased the tenderness of the cake when compared to the control.

The cereal brans were carried through a stepwise separation method to measure individual dietary fiber components. These included water soluble hemicellulose, water insoluble hemicellulose, cellulose, pectin, and lignin. Wheat brans were similar in content, showing values of approximately 0.8% pectin, 26% water insoluble hemicellulose, 8% cellulose, and 4% lignin. The other brans varied widely. Oat hulls were lowest in most fiber components. Soy bran was the only bran containing a substantial quantity of pectin. It was lower than the wheat brans in water insoluble hemicelluloses but contained the largest quantity of cellulose. Corn bran insoluble contained the largest quantity of water hemicellulose.

Statistical programs were used to calculate prediction equations for cake parameters using fiber components as the independent variables. Fiber components could be used to predict the following parameters: tenderness, volume, viscosity, cell size, cell wall thickness, and grain. The hemicelluloses appeared to have a large effect on cake quality while pectin and cellulose at the levels used had little effect on most parameters. Lignin often appeared in the prediction equations as an interaction with a hemicellular component.

To Brian and Tesla for his help and her hindrance

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INTRODUCTION

Recent concerns over the need for dietary fiber in the human diet have led to extensive research on the physiological effects of the various fiber components. Less work has been conducted on the methods of incorporating fiber into the diet. Most of the work has been aimed at the bread system.

Work with the cake system has shown that levels up to 30% (based on the weight of the flour) soft red wheat bran could be successfully incorporated into white layer cakes, although several objective and sensory attributes were significantly altered. However, no attempt was made to determine which dietary fiber components were responsible for the change in cake parameters noted.

Fiber components are known to behave differently in the prevention of disease and show interactive effects. While one component may be important in the prevention of one disease, it may not be important in another. Dietary fiber components could be expected to differ in their effects on cake parameters. Also, different fiber sources could be expected to function differently due to varying levels of fiber components and possible interactive

effects.

This study was conducted to test the validity of these assumptions. Various varieties of wheat brans as well as corn, oat, and soy bran were incorporated at a level of 30% of the flour to test the hypothesis that brans from different sources would affect cake quality in different manners. The dietary fiber components in each bran were analysed in an effort to pinpoint which components were responsible for the changes in cake parameters noted.

The only work with individual fiber components in various baked systems has been done using the water soluble and insoluble hemicelluloses of wheat endosperm. To analyse the fiber components, it was necessary to revise existing methods of fiber measurement. Most existing methods measure only total fiber content. The methods which do break down fiber content into the individual fiber components are incomplete and have inherent difficulties. The analysed component levels were used as the independent variables to see if the variations in cake parameters could be predicted by fiber component levels.

Finally, a series of cakes were made incorporating individual fiber components in the form of cellulose and derivatives coated with CMC and pectin to analyse the effects of high levels of individual fiber components.

REVIEW OF LITERATURE

Dietary Fiber

"Fiber" is a general term, denoting food components which are not digested by the human digestive system. Over the years it has been defined and measured in a number of ways, due to the diversity of compounds which can be included in the term "fiber". Today it is generally accepted that fiber components include cellulose, hemicellulose, pectin, lignin, as well as waxes and mucilages which may be associated with the cell wall.

Nutritional Aspects of Fiber

It has been known for many years that certain fiber constituents are helpful in disease prevention. As early as the 1930's, apples were being prescribed for the treatment of diarrhea, and pectin was recognized as the component responsible for this effect (Malyoth, 1934). Fiber was also believed to have a detoxification effect in the body by precipitation of heavy metals (Stuewer and Olsen, 1940; Murer and Crandall, 1942). However, the need for fiber in the human diet has been generally ignored. Recently, much attention has been focused on the

nutritional value of fiber. This was brought about mainly due to the findings of Denis Burkitt and Hugh Trowell. Through epidemiological data, they have related the incidence of diseases such as diverticulosis, colonic cancer, appendicitis, hernia, vericose veins, and heart disease to a low dietary fiber intake (Burkitt, 1973; Trowell 1972; Burkitt 1971a; Burkitt 1971b). The lack of fiber has been hypothesized to cause disease by decreasing stool bulk, thereby increasing transit time, intralumenal pressures, and concentration and reaction time of toxic substances (Burkitt 1971b).

Recently there has been a concentrated research effort to determine if any or all of these diseases are caused by a lack of fiber in the diet, and if so, which components are responsible. Extensive reviews on the subject have been written (Spiller and Amen, 1975).

To date, the results of these studies are as follows. Fiber does increase the water holding capacity of the feces (Williams and Olmstead 1936; Findlay et al. 1974), however, fibers from different sources and more specifically the various fiber components differ in their water holding capacities (Eastwood, 1973; McConnel et al. 1974). Of the various fiber components, pectic substances and hemicellulose have a high water holding capacity, while cellulose is moderate, and lignin is minimal in it water holding capacity (Eastwood 1973).

It has generally been found that increasing the amount of fiber in the diet does increase stool bulk and decrease transit time (Eastwood et al. 1973; Payler et al. 1975; McCance et al. 1953). It has been hypothesized that changes in fecal bulk and transit time may be due to the production of volatile fatty acids by the microbial decomposition of fiber components in the large bowel (Cummings, 1973).

It is believed that high serum cholesterol levels are related to an increased risk of cardiovascular disease (Anon, 1970). Several investigators have found that various fiber components can lower cholesterol levels. Pectin has been found to be particularly effective (Leveille and Sauberlich, 1966; Tsai et al. 1976). The mechanism of cholesterol reduction may be due to an inhibition of bile acid reabsorption (Leveille, 1975). However, controlled research studies have shown many discrepancies, therefore the hypothesis has not been substantiated (Cummings, 1973).

The acidic polysaccharide components of fiber have been found to bind metals and have the ability to act as cation exchange resins due to free carboxyl groups (Eastwood, 1973). What effect this may have in the gut is not known. As postulated in the 1930's, fibers have been found to reduce the toxic effects of chemicals in rats (Ershoff and Marshall, 1975). However, the effectiveness

depended on the type of fiber used.

Location of Fiber Components

Figure 1 shows the location of the fibrous components in the cell. Cellulose and hemicellulose are located in the primary and secondary cell walls. Cellulose concentration is greater in the secondary cell wall, while hemicelluloses are concentrated in the primary cell wall. Lignin increases from the middle lamella to the secondary cell wall, while the highest concentration of pectin is in the middle lamella.

Structure of Fiber Components

<u>Cellulose</u>. Chemical studies on purified cellulose (Figure 2) indicate that it is a polyglucosan, and that it is improbable that a substantial amount of other substances are present. The position of the bonds linking the glucose molecules has been determined through methylation studies to be a linear 1,4 linkage, and acetolysis studies have indicated that this linkage is β -D-(1 \rightarrow 4) (Whistler, 1963; Hulme, 1970). It has also been found that the glucose molecules are in the pyranose form (Pigman and Horton, 1970).

Cellulose chains bond to each other through hydrogen bonding of the free hydroxyl groups. This creates fibrils of great strength. Cellulose chains align to form microfibrils. The microfibrils in turn bond to form

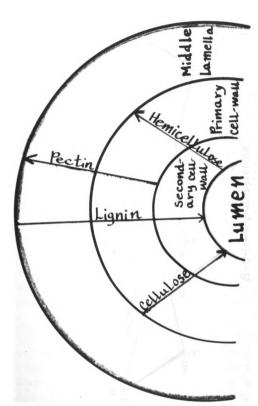
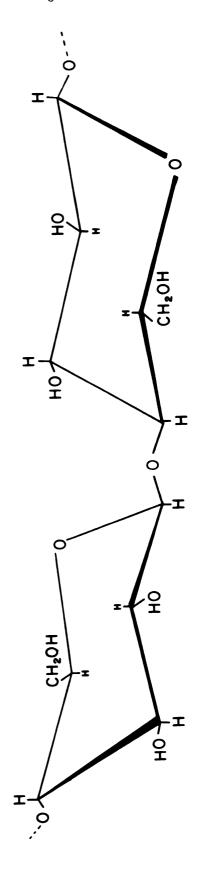


Figure 1. The location of fiber components in the cell.



 β -D-(1+4) glucopyranose

Figure 2. The structure of cellulose

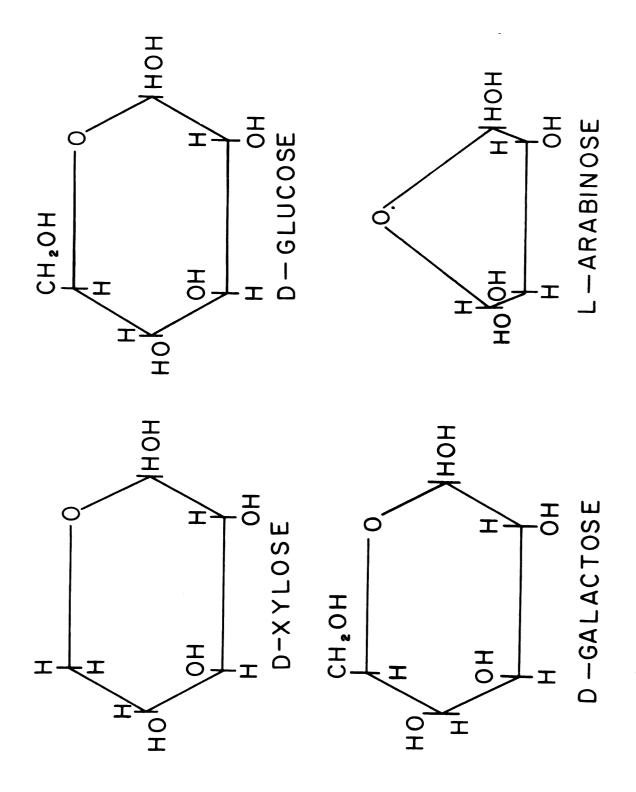
fibers. Disordered regions with low hydrogen bonding occur at irregular intervals. These amorphous regions are more susceptible to attack (Pigman and Horton, 1970).

Although purified cellulose is known to have few irregularities in structure, in its native state it has been found to be closely associated with hemicellulose and lignin. There is some evidence that cellulose may be bound with hemicellulose (Pigman and Horton, 1970), and lignin has been found in the cellulose fraction of cranberry pulp (Pigman and Horton, 1970).

Hemicellulose. Hemicelluloses are polysaccharides which in the plant in close association cellulose. They were at first believed to be precursors synthesis. Most hemicelluloses in cellulose heteroglycans containing between two and four sugars (Figure 3). The most common glycans include L-arabino-Dxylans, L-arabino-D-gucurono-D-xylans, 4-0-methyl-Dglucurono - D - xylans, L-arabino - (4-0-methyl-D-glucurono) - Dxylans, D-gluco-D-mannans, L-arabino-D-galactans, and α and β glucans. These glycans are usually branched and partially acetylated (Pigman and Horton, 1970).

Xylans are the most common hemicelluloses occurring in all land plants. The most common single unit side chains of these are L-arabino furanose linked $1 \rightarrow 3$ and D-glucopyranosyl uronic acid linked $1 \rightarrow 2$. Side chains may be on connecting or separated xylose units. Cereal

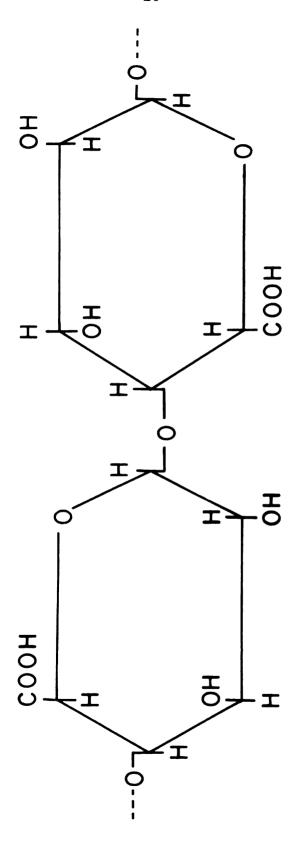
Figure 3. The common sugars found in hemicelluloses.



D-xylans have been found to contain both sugars as side chains, whereas legumes are slightly higher in uronic acids, and have no arabinose (Pigman and Horton, 1970). The xylans also differ in arabinose/xylose ratio, degree of branching, and associated protein.

Pectin. The backbone structure of pectin is $\beta-D-(1 \rightarrow 4)$ galacturonic acid (Figure 4). Many pectins have neutral sugars covalently linked to them. These include xylose, rhamnose, galactose, arabinose, and glucose. Arabinose, galactose, and xylose have been found to be attached as side chains, while rhamnose-rich blocks may link galacturonan chains. The carboxyl groups of galacturonic acid are partially methylated and the secondary hydroxyls may be acetylated. The sugars which have been found side chains indicate that pectin is chemically bonded to other cell wall constituents - notably hemicellulose (Hulme, 1970).

Lignin. Lignin cconsists mainly, or entirely of phenylpropane building stones. It also has a hydroxyl or methylated hydroxyl group para to the propane side chain. It may have up to two methoxyl groups at positions ortho to the phenolic oxygen function. Individual phenyl propane groups are bonded by ether and carbon-to-carbon bonds (Figure 5). These latter type bonds are extremely resistant to chemical degradation. Ether linkages may



 $\alpha - D - (1-4)$ galacturonic acid Figure 4. The structure of pectin.

Figure 5. Examples of possible lignin linkages (taken from Schubert, 1965).

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ÒН

exist at more than one point. Although chemical proof of many of these linkages is lacking, some support has been lent through biochemical studies on the formation and degradation of lignin (Schubert, 1965).

Composition of Cereal Pentosans

One of the earliest investigations of the water soluble pentosans of wheat was conducted by Perlin (1951a). He found that this fraction contained anhydro-L-arabinose and anhydro-D-xylose residues. Galactose was also found which could have been separate or part of a pentosan-galactan. A great deal of similarity was found between the sugar composition of different flour varieties, however molecular size of pentosan fractions did differ.

Another early study by Perlin (1951b) investigated the effect of graded hydrolysis on the pentosan fraction. Anhydro-L-arabinose was preferentially removed, leading to an insoluble residue of anhydro-D-xylose residues. A pentosan containing 35% or more arabinose was soluble in cold water. Methylation studies demonstrated the presence of 2,3,5-trimethyl-L-arabofuranose, 2,3-dimethyl-D-xylose, 2-methyl-D-xylose and D-xylose in ratios of 3:3:1:1.

Methylation studies in addition to periodate oxidation led Perlin (1951b) to propose the structure shown in Figure 6. Further studies have fractionated



Figure 6. Structure proposed by Perlin (1951b) for hemicellulose.

water soluble pentosans on a DEAE cellulose column. Kundig et al. 1961) used this technique to investigate protein and galactose in order to determine whether these constituents were bound to the pentosan fraction. five fractions. The first obtained was a pure arabinoxylan. Fractions 2 through 5 contained galactose and protein, however fraction 3 contained no xylose. They concluded that protein was covalently bound to most of the fractions as a glyco-protein. A further study by Holme (1962) using electrophoretic and ultra centrifugal sedimentation behavior has shown that the pentosan fraction containing 5% protein is a homogenous fraction.

The glycoprotein fracton 2 has been further studied by Neukom et al. (1967). They treated this fraction with pronase, liberating two fractions. One fraction was a pure arabino-xylan and the second an arabinogalactan. They concluded that galactose is not linked to the xylan chain, but is part of an arabino-galactan and is most likely connected to the arabinose of the arabinoxylan via a polypeptide bridge. Possible linkages were hypothesized as 1) an ester bond between carboxyl groups of an amino acid residue and the secondary hydroxyls of xylose or 2) O-glycosidic linkages between the reducing end group of xylose and the hydroxyl groups of serine or threonine.

Fincher and Stone (1974), through fractional

precipitation of water soluble components, were able to separate out a high molecular weight arabinoxylan and a low molecular weight arabinogalactan. The latter was associated with a hydroxy proline rich peptide which was believed to be covalently bound to the arabinogalactan. They suggested that some of the protein eluted and measured by Kündig et al. (1961) is free. This has also been found by Lineback et al.(1977). Other work of Preece and Hobkirk, cited by Fisher and Stone (1974), suggests that galactose rich polysaccharides may be widely distributed in cereal grains.

Several workers have studied the water soluble pentosan compositions of various wheat varieties. Medcalf et al. (1968) found that durum (Wells) pentosans contain a higher proportion of arabinose and a more highly branched stucture than a hard red spring wheat (Selkirk).

D'Appolonia and MacArthur (1975) studied various varieties of conventional and semi-dwarf hard red spring wheats. Differences did occur between varieties, but classes of wheat could not be differentiated by any particular measurement.

Lineback et al. (1977) examined fractions from a hard red winter, hard red spring, and soft red winter wheat. The carbohydrate composition varied, particularly in fractions 2, 3, and 5, however, differences were again not sufficient to separate the pentosans by source. Table

1 compares the sugar composition of fractionated pentosans obtained by several investigators.

The composition of the water insoluble hemicelluloses has also been studied. Montgomery and Smith (1955) found the hemicellulose to contain D-xylose (59%), L-arabinose (39%), and D-glucose (2%). Through methylation studies they determined that the stucture was similar to that of the water soluble hemicelluloses.

Cole (1967) isolated these hemicelluloses and separated fractions on DEAE-cellulose. He found a total sugar composition of 54% xylose, 33% arabinose, 11% glucose, 2% galactose, and 2% protein. Fracton 5 was found to include the protein and was believed to be a glycoprotein. Whether glucose was covalently linked to the pentosan fraction was not known. They found few if any carboxyl groups attached to the polysaccharide molecules, even though they showed substances with a similar Rf to uronic acids. A further study by Cole (1969) on fraction 1 of the hemicelluloses found the existance of approximately 1% acid, and Whistler and Smart (cited by Cole, 1967) have found acid in nearly all hemicellulose preparations.

Comparisons of water soluble to insoluble pentosans have shown both a similar degree of branching (Medcalf et al. 1968) and a lower degree of branching in the insoluble pentosans (D'Appolonia and MacArthur, 1975).

The latter researchers also found that water insoluble pentosans contained fewer glycoproteins and a higher intrinsic viscosity than the water soluble hemicelluloses. They found no galactose present.

Work on corn fractions have shown that none of the fractions contain pectin, although the pericarp contains a polyuronide hemicellulose with as much as 12% uronic acid. Sugars found in the pericarp included xylose, arabinose, galactose, and uronic acids (Seckinger, et al. 1960).

Early work on the water soluble gums of different cereals (Preece and Mackenzie, 1952) showed that rye is the best source of pentosans, wheat is intermediate, and oats and corn are poor. Corn was high in total water solubles due to high amounts of starchy materials. It was also hypothesized that the bran of wheat may contain more water soluble pentosans than the endosperm.

Alkali extraction of wheat bran by Adams (cited by Hlynka, 1964) has shown it to contain arabinose (59%), xylose (38.5%), and glucuronic acid (9%). Other studies have shown xylose, arabinose, galactose, glucose, glucuronic and 4-0-methyl glucuronic acid in bran hemicelluloses. Studies of bran have shown it to be more branched than flour (Hlynka, 1964). Legume pentosans have been found to contain more uronic acids than cereals, but no arabinose (Pigman and Horton, 1970).

Table 1. Carbohydrate composition (% of total) of acid-hydrolized fractions eluted from chromatography on DEAE cellulose¹

					In	vest	igat	or ²			
	Fraction	1	2	3	4	5	6	7	8	9	10
I	Arabinose Xylose Galactose Glucose	37 62 1 0	37 63 0	40 60 0	- - -	43 49 0	45 55 0	40 60 -	36 64 -	4 1 5 9 -	40 60 -
II	Arabinose Xylose Galactose Glucose	43 ³ 27 30 0	44 ³ 40 16 0	443 49 7 0	43 49 8 0	41 42 17 0	36 38 26 0	50 50 -	45 55 -	47 53 -	47 53 -
III	Arabinose Xylose Galactose Glucose	50 ⁴ 8 42	33 ⁴ 12 54	38 ⁴ 5 57 -	40 21 39	35 0 65	46 0 54	50 35 - 15	50 45 5	32 17 51	37 7 56
IV	Arabinose Xylose Galactose Glucose	42 6 52	41 6 54	36 2 61	38 6 56 -	43 0 57	43 8 49	53 11 - 37	50 10 40	32 8 60	30 9 61
V	Arabinose Xylose Galactose Glucose Mannose	26 38 7 27 2	21 23 10 42	8 25 7 48 12	22 31 0 47 0	- - - -	17 81 2 0	43 39 - 17	42 46 12 -	42 58 - -	44 56 - -

¹Taken from Lineback et. al 1977.

²1,2,3,from Lineback et. al. 1977 from Scout R-70, Chris, Logan flours; 4 from Lin and Pomeranz (1968) from HRS (Marquis); 5 from Wrench (1965) from Australian bakers flour; 6 from Kundig (1961) from HRS (Manitoba); 7,8,from Medcalf et. al. (1968) from durum (Leeds) and HRS (Thatcher); 9,10,from D'Appolonia and McArthur (1975) from HRS (Justin and World Seeds 1809).

 $^{^3}$ Values from fractions 11_a and 11_b averaged.

 $^{^{4}}$ Values from fractions 111 $_{a}$ and 111 $_{b}$ averaged.

Separation and Quantitation of Fiber

Many research studies have analysed individual percentage of the major carbohydrate The portions of wheat endosperm and bran are shown in Table 2. pentosans of wheat endosperm have been subdvided into soluble and insoluble fraction. Crude water solubles have been reported as being between .55 and .81% of the total flour. After treatment with α -amylase was reduced to .44-.55%. The water solubles were further estimated to contain 50-68% carbohydrate and 8-22% protein (Lineback et al. 1977). In addition, many separation schemes have been devised over the years for analysis These include both dietary components. physical and chemical separations. Physical separation methods been used where chemical modification is undesirable, for example in baking studies where components are transfered from one flour to another or for sugar analysis of an individual component. Chemical separation methods generally been used for component quantitation.

Physical Separation Methods

Physical separation methods for obtaining water soluble pentosans usually include mixing the material in a Waring blender and then centrifugation. The supernatant may be heated to inactivate enzymes. It is then usually treated with amylase and dialysed to remove soluble

Carbohydrate and lignin contents of flour and brans

Table 2.

Source	Hemicellulose	Cellulose	Starch	Lignin	Reference
Wheat Endosperm	7 ← N 2 · · · · · · · · · · · · · · · · · · ·	.6;.8	67.4	.03	Fraser et. al. 1956 Fraser and Holmes 1957 Southgate 1978
Wheat Bran unknown	26.2 26.5 22.4	21.4	8.6 7.5 9.0		Fraser and Holmes 1957 Pomeranz 1971
HRS I II		12.0 12.4		w w 4 · w	Rasper 1978
HRW		0.6		3.1	Ξ
SMS		6.6		1.7	=
I MMS		8.8 6.6		2.4 2.1	Σ :
Soy Hulls		9.04		3.3	Ξ
Oat Hulls		35.0		6.7	=
Corn Bran II III III	47. 58. 67.	22.7 14. 19.		9.7.3.0	shaller 1977 "

starch. Proteins may be removed by the addition of tricloroacetic acid or by heat.

To bring about a more complete separation of flour components, Donelson and Wilson (1960a) used the following scheme. The flour was mixed with water to effect maximum gluten development. The gluten was then removed by screening and washing. The filtrate from the screen was centrifuged and a water soluble layer decanted. A layered residue remained which was physically separated into a top layer which contained starch tailings and a bottom layer which contained prime starch.

Chemical Measurement of Fiber

The first method devised to quantitate fiber content was crude fiber (CF). This method was developed in the 1800's and values are still found in food composition books. This method involves hydrolysis with .255 N sulfuric acid followed by .313 N sodium hydroxide. Approximately 80% of the hemicelluloses, 50 to 90% of the lignin and 20 to 50% of the cellulose is degraded and therefore not measured (Schaller, 1977). The xylan fraction of the hemicellulose tends to remain with the crude fiber.

Several methods have been devised which utilize detergents to separate dietary components. The acid detergent fiber (ADF) method was developed for the estimation of the nutritive value of fibrous feeds by

Van Soest (1963). This method used cetyl trimethylammonium bromide in an acid solution to dissolve proteins yielding mostly cellulose and lignin. The value obtained was related to the indigestible portion of the feed. Van Soest chose this method for the removal of proteins because protein must be removed to obtain good lignin values, and akali dissolves part of the lignin. He did find, however, that the detergent solubilized part of the hemicellulose as well as the protein.

This method was altered by Baker (1977). He used a less corrosive solvent for the detergent, thereby obtaining higher recovery values for lignin and cellulose.

Van Soest developed a neutral detergent fiber method in an effort to more completely analyse substances which are only partially available to animals (Van Soest and Wine, 1967). A 3% solution of sodium lauryl sulfate buffered at pH 7 was found to remove lipids, water soluble materials, starch, soluble protein and pectin. The residue included protein which was attached to the cell wall, hemicellulose, cellulose, lignin, heat-damaged protein, kerratin, and lignified nitrogen compounds. Hemicellulose content was estimated by the difference between neutral and acid detergent fiber values.

This method does cause some losses in hemicellulose, since anionic detergents dissolve some of the polysaccharide fractions at all pH ranges. However,

recoveries were greatest between pH 6-7 (Van Soest and Wine, 1967).

The final version of the detergent methods developed enzymatic neutral detergent fiber was (Robertson and Van Soest, 1977). This utilizes α -amylase prior to extraction, due to the and proteolitic enzymes filtration difficulties which had been previously encountered. However, losses of water soluble insoluble hemicelluloses and pectins still occur. Results given by the various detergent methods are shown in Table 3.

Enzymatic methods for the analyses of fiber constituents have been used for many years. These make an effort to create a fiber residue which is similar to that created in the body through the use of proteolitic and diastatic enzymes. These include methods of Weinstock and Benham (1951), Remy (1931), Williams and Olmstead (1935), and Hellendoorn et al. (1975). Enzymatic methods again do not analyse water soluble fiber components and give only one dietary fiber value.

There have been many schemes devised over the years for the analyses of separate fiber constituents. Many of these have been devised for woods which are high in lignin components. They generally involve delignification with sodium chlorite to yield holocellulose, and solubilization of hemicelluloses with potassium hydroxide (Wise et al.,

Table 3. Dietary fiber content of materials measured by acid detergent fiber (ADF), buffered acid detergent fiber (BADF) and neutral detergent fiber with enzymes (NDF + Enz)

Sample	ADF %	BADF %	NDF + En
Wheat Bran ¹			
HRS 1	15.4	23.8	44.5
2	15.7	24.1	45.4
HR W	12.1	20.0	38.6
SWW 1	10.8	17.2	38.2
2	10.7	16.3	38.2
Wheat Bran ²	-	-	36
Corn Bran ¹	23.3	37.6	93.7
Corn Bran ²	_	-	8 9
Oat Hulls 1	41.7	76.2	88.6

¹From Rasper (1978).

²From Schaller (1977).

1946). Different hemicellulose fractions can be separated by differing the strength of the potassium hydroxide solution. Although the concentration was found to be arbitrary, 5% and 24% solutions have been most often chosen. Twenty four percent KOH has been chosen as the last extractive because most of the cellulosans are extracted, and further extraction removes very little carbohydrate material (Wise et al. 1946).

With this method, separations are incomplete losses of fractions do occur. Delignification with sodium chlorite leads to losses in the carbohydrate fraction. These losses can bе minimized bу incomplete delignification, leaving 2-4% of the lignin unextracted (Wise et al. 1946). The degradation of hemicelluloses has been found to be due to the oxidation of the reducing to aldonic acids and slight depolymerization and ends oxidation of the 2,3 glycol groups (Pigman and 1970).

The alkaline solutions used to extract the hemicelluloses can cause extensive degradations. If oxygen is present, cellulose is degraded. Even under an oxygen free atmosphere, xylans are slowly degraded via a β -elimination reaction, and uronic acid residues may also be destroyed (Pigman and Horton, 1970).

The α -cellulose fraction obtained is not entirely free of furfural-yielding material. These may be due to

xylose or uronic acid residues of the hemicellulose fraction. Also, even when complete delignification was tried some lignin was found in the hemicellulose fraction (Wise et al. 1946).

In this method, pectin is not isolated, but is a part of the hemicellulose fraction. It is primarily found with hemicellulose A. To examine pectin and soluble hemicelluloses as a separate fraction, boiling water extractions have been employed (Jermyn and Isherwood, 1956).

Another variation of this scheme was tried by Thornber and Northcote (1961). They extracted soluble material with boiling water for 3 hours and extracted the fat with ethanol-benzene. The residue was treated with sulfuric acid to yield a residue of klason lignin (low molecular weight lignin which is soluble in some solvents). The supernatant was extracted with EDTA and solubilized pectic substances precipitated with 80% ETOH. The residue was then delignified and the holocellulose further treated to separate hemicellulose and cellulose.

The second main scheme for the separation of fibrous components uses fat solvents, enzymes, and varous concentrations of acid to remove starch, protein, hemicelluloses, and cellulose, leaving lignin as an end product. One such scheme was used in 1929 by McCance and Lawrence to evaluate the food value of vegetable

carbohydrates. Fiber was defined as the material not extracted by warm water, diastase, and 1-3% boiling acid. It included cellulose, oxycellulose, lignin, and anhydrides of the pentoses. Another such method was employed by Ellis et al. (1946) to separate lignin from protein containing plant material. He utilized ethanol, benzene, pepsin, 5% sulfuric acid, 72% sulfuric, and then 3% sulfuric acid yielding lignin as a residue.

More recenty, Southgate (1969) developed a similar scheme for the analysis of fiber components. He extracts sugars with methanol, and water solubles with hot water. He then solubilizes the starch from the soluble and insoluble residue with takadiastase and adds ethanol to precipitate hemicelluloses. Five and seventy two percent sulfuric acid remove hemicellulose, and cellulose respectively, leaving lignin as a residue. Results of his analysis are shown in Table 4.

These schemes, as any other which can be developed for the separation of several fiber constituents is imperfect due to the effect that one chemical may have on several components, and the fact that many fiber components are chemically bonded causing incomplete extraction. Linkages between pectin and xylans are incompletely hydrolyzed by acid. Aldobiouronic acid groups are formed (McKee and Dickey, 1963).

Lignin cannot be isolated in a pure form, for it is

Total dietary fiber and its composition in various wheat products $^{\mathsf{1}}$ Table 4.

		Composition of the dietary fiber	the dietary %	fiber	Cellul Cellul	osition o osic Poly	Composition of the non- Cellulosic Polysaccharides
Sample	Total Dietary Fiber ² (g/100 g)	Non-cellulosic	Cellulose	Lignin	Hexose	Pentose	Uronic Acid
White Flour (72%)	3.45	80	19	٢	80	11	6
Whole Meal Flour	11.0	7.2	20	∞	39	8 #	13
Bran	0.84	η Δ	18	7	19	69	12

¹Taken from Southgate (1977)

 $^2\mathrm{On}$ a dry weight basis

insoluble in any solvent which does not react with it chemically (Pearl, 1967). Lignin condenses readily with proteins under acidic conditions, if these are not removed by enzymes and acid treatment (Sarkanen and Ludwig, 1971; Brauns, 1952). The products formed have little relation to the original protein, so nitrogen cannot be measured and multiplied by 6.25 to subtract out the protein content (Brauns, 1952). Up to one to two percent of the total monomer bonds in lignin are probably bound with hemicelluloses. These may not be completely hydrolyzed (Sarkanen and Ludwig, 1971). It has also been found that up to 10% of the lignin in wood may be low molecular weight polymers which are released through the hydrolytic processes associated with aging. These will dissolve solvents such as ethanol and are not analysed (Sarkanen and Ludwig, 1971).

Pectic substances have generally not been measured as a part of these schemes. Separate separation schemes have involved the use of enzymes such as pectinase (McComb and McCready, 1952), titration (Whistler and Wolfrom, 1962), extraction with ammonium oxylate (Southgate, 1976b), or decarboxylation.

Fiber Quantitation

Quantitation of separated fiber fractions can be done gravimetrically or chemically. Chemical analysis relies on measuring major sugar components colorimetrically,

through thin layer chromatography or gas liquid chromatography, or through formation and quantitation of products such as CO_2 . Several colorimetric methods have been proposed for the measurement of sugars, however many of these have the drawback of being nonspecific.

Hexoses may be measured by the anthrone reaction. This minimizes the interference by pentoses, hexuronic acids, and 2-deoxypentoses. The blue color that develops shows a maximum near 625 nm for hexoses and 6-deoxyhexoses. Pentoses develop a yellow color with an extinction coefficient of only a few percent of that of hexoses (Whistler and Wolfrom, 1962). Uronic acids do not interfere (Helbert and Brown, 1956).

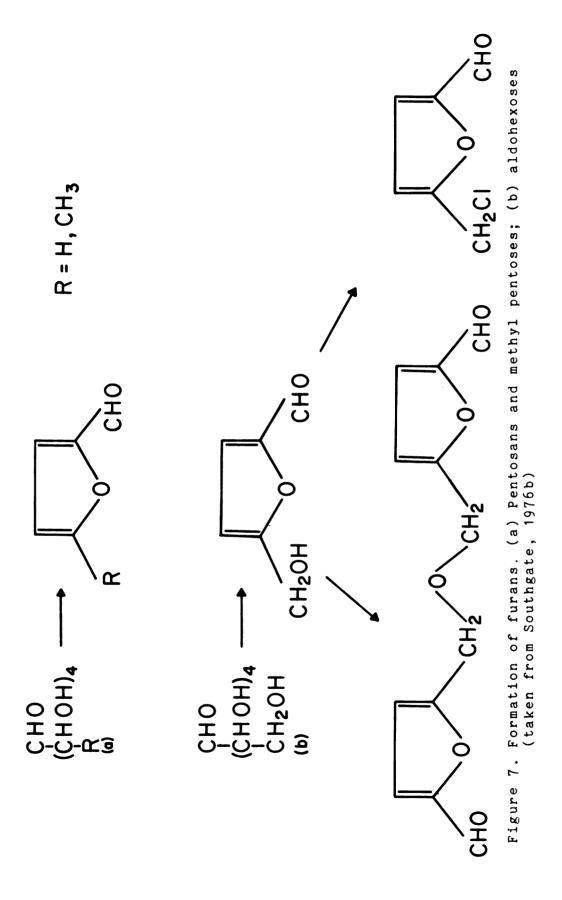
Hexuronic acids can be measured by carbazole and sulfuric acid. This reaction is based on the formation of 5-carboxyl-2 formyl furan (Stutz and Deuel, 1956). However, not all of the uronic acids show the same extinction coefficient. The absorption of galacturonic acid was found by Dische (1947) to be 21.5% weaker than glucuronic acid. Polyuronides showed absorptions equal to or greater than the monomer. Hexoses and pentoses form different colors, however, the interference on an equivalent basis is 5-7% for glucose and 1% for pentoses based on D-glucuronic acid (Whistler and Wolfrom, 1962).

Bial's orcinol reaction for pentoses measures all adopentoses equivalently and ketopentoses also show an

absorption maximum at the same wavelength. However, this reaction is not specific. 6-deoxyhexose, hexuronic acids, heptoses, trioses, and in high concentrations D-mannose, and D-galactose all show absorption maxima in the same range (Whistler and Wolfrom, 1962).

All of the color reactions are based on condensation reactions which a monosaccharide will undergo when in the presence of a strong acid and phenolic and other substances. Pentoses and methyl pentoses form 2-furfurals while aldo hexoses form 5(hydroxy-methyl)-2-furaldehyde (Figure 7).

Sugar measurements through thin layer chromatography involve sugar hydrolysis, separation, and measurement by use of a densitometer or elution and color measurements. Gas liquid chromatography also requires sugar hyrolysis, however, derivatives such as trimethylsilyl or alditol acetates must be made before column separation. Both of these methods allow individual sugar quantitation. Uronic acids can further be measured through titration of the carboxyl group or measurement of carbon dioxide evolution.



Cake Structure

A cake is a complex chemical system consisting of a variety of ingredients which interact in the presence of water and heat to form the typical cake structure. The way in which each component reacts in the system is dependent upon the type and level of the other components.

Two types of cake formulas (high and low ratio) are commonly made. Both include as ingredients low extraction soft wheat flour, sugar, shortening, salt, liquid, whole egg or egg white, leavening agents and flavorings. They differ in sugar level, type of shortening necessary, and method of mixing. These ingredients are mixed, forming the cake batter which is an oil in water emulsion containing four distinct phases (aqueous, fat, vapor, and solid starch). Interactions can occur at the interfaces of the various phases. During baking the batter must be stable enough to prevent phase separation (Howard, 1972).

Three components have been found to be necessary for adequate batter stability of the high ratio cake. These include soluble proteins, polyvalent cations, and surface active lipids. These interact at the oil and water interface, forming a film around each fat droplet, thereby preventing interference with the foaming properties of the soluble proteins. These emulsifying agents have also been reported to contribute to the formation of a fine grained cake. Mono and diglycerides aid in the dispersion of fat

in the aqueous phase. Emulsifiers which are α tending or anionic lipids aid in batter stability by forming at the oil/water interface (Howard, 1972).

Many of the ingredients commonly incorporated cakes are non-essential. Howard (1972) found that a normal cake structure could be achieved using egg protein, wheat starch, stearic acid (anionic lipid), CaCl (polyvalent ion), leavening agent, and water. Polyvalent cations are normally supplied through flour, egg, milk and leavening agents. The type of starch used was found to be critical due to its inherent temperature gelatinization. Rice starch could not be used because its gelatinization temperature was never reached during baking.

Since the cake system is so complex, the level of an ingredient and ingredient interactions can have a profound effect on the ultimate product. Water concentration has been found to have a critical effect on the extent of starch gelatinization and ultimate crumb structure (Wilson and Donelson, 1963). The level of water needed is altered by the absorption characteristics of the flour used and other ingredients which may be present. Ingredients such as milk solids, egg proteins and sugar have definite water requirements. These materials are more hydrophilic than starch, therefore, water bound by these ingredients is not available for starch gelatinization.

Too little water leads to a cake with a sunken center and coarse cells with thick cell walls. Optimum water levels yield cakes with a rounded contour, good volume, and small, uniform cells with thin cell walls. Too much water causes excess gelatinization. The cake is peaked and internal structure takes on a gel-like character. The water requirements of cakes with flour as the only protein source versus cakes containing egg and milk proteins have been compared (Wilson and Donelson, 1963). Although the added proteins increased the water requirements, the water level was less critical indicating that proteins may act as a buffer, moderating the response to liquid.

Studies by Kissel and Marshall (1962) have found that baking powder and sugar/water ratios are critical to volume and contour, however, shortening level had little effect. Howard's (1972) study verified these results.

Functionality of Flour Fractions

Cake Systems

Cake flour is made up of a variety of components including soluble and insoluble proteins, starch, soluble and insoluble hemicelluloses, cellulose, and lignin. Fractionation procedures have been developed which separate components into the following fractions: water solubles, starch, tailings (containing the water insoluble hemicelluloses) and gluten. The majority of the protein

was found in the water soluble and gluten fractions.

The separated fractions have been interchanged different ways to study the effect of each in combination with various levels of the other fractions. Donelson Wilson (1960b) interchanged fractions of a high and a low quality flour. They concluded that gluten, solubles, and tailings interact to determine the quality of the basic cake structure. The quality of each fraction differed depending on its source. For instance, lower levels of gluten from the high quality flour than from the low quality flour could be used to form high quality cakes. It appeared that protein solubility was involved a more soluble protein could be used at lower levels and still produce a high quality cake.

Starch was not involved in interactions with other components although quality again varied depending on the source. A slight increase in starch concentration over the normal level increased cake volume. Although the starch from the low quality flour was found to be superior, its effect was minimized by the poor quality of the other flour components.

Studies which have altered the level of components of the same flour (Baldi et al. 1965) have shown that an increase in tailings concentration at the expense of prime starch improves volume and internal characteristics. The resulting cake has a fine, silky texture and is more moist

and tender. The granules of starch are smaller and thinner, indicating that tailings interfere with starch-starch bonding. This increase in tailings concentration is accompanied by an increased water requirement.

Lower levels of tailings are needed in the high ratio cake as compared with the low ratio cake. This is believed to be due to an interaction between tailings and sugar to form a more stable gel-like batter. hypothesis has been substantiated by results of amylograph viscosity studies on starch, in which hot paste viscosity and gel strength increased with an increase in sugar when tailings were present (Baldi et al. 1965). Increased levels of sugar usually lower hot paste viscosity and gel strength by competing with the starch for water (Bean and Osman, 1959). Hemicellulose may also be involved binding with other flour components, since batters containing tailings were less curdled and showed less separation of the aqueous and solid phases (Baldi et al., 1965).

When gluten or starch levels were altered slightly, little volume effect was noted. However, large changes decreased volume substantially. An increase in water solubles tended to decrease volume slightly. This effect was essentially linear with concentration (Donelson and Wilson, 1960a).

Donelson and Wilson (1960a) also found that differences in cake volume could not be attributed to protein level, since cakes containing a wide level of protein were of equal volume.

Studies on the water binding capacity of water soluble hemicelluloses have found that there is no single value, but depends on the available water, speed of mixing and other flour components which may be present (Jelaca and Hlynka, 1971). Water soluble and insoluble pentosans have been found to absorb 5.3 and 6.1 times their weight respectively (14% moisture) when measured alone, however, when gluten was present absorptions of water soluble hemicelluloses dropped to 4.8.

Breads and Cookie Systems

In a bread system, the addition of water solubles has generally been found to increase loaf volume (Pence et al. 1951; Cawley, 1964), however, some varietal differences have been noted (Jelaca and Hlynka, 1972). This effect has been found to be due to the pentosan fraction rather than soluble proteins (Cawley, 1964; Wrench, 1965; Tracey, 1964). The effect of water insoluble pentosans is more debatable. In some cases they have been found to increase loaf volume while causing a deterioration in internal characteristics (Jelaca and Hlynka 1972). In other cases a decrease in loaf volume was noted (Kulp and Bechtel, 1963).

It is believed that these effects are due to protein-polysaccharide interaction. Udy (1957) found that mixtures of gluten and water soluble polysaccharides will in dilute acid. The extent of the interaction gel depended on the molecular size of the polysaccharide. believed that weak secondary forces such as electrostatic Studies in which water soluble forces were involved. pentosans have been found to increase the resistance of gluten to extension also point to а possible protein-polysaccharide interaction (Jelaca and Hlynka. 1972).

Cawley (1964) added many gums to the bread found that the type of gum was important, since many gums did not increase bread volume. Solubility, number, and size of side chain substituents were more important. Soluble carboxy methyl cellulose (CMC) was effective while insoluble CMC was not. Also, the greater the degree of the effect. branching. the greater Charged polysaccharides such as pectin were in general ineffective. He therefore concluded that hydrogen bonding rather than electrostatic interactions must be involved.

Water soluble and insoluble hemicelluloses have been found to differ in molecular shape and degree of branching (Tao and Pomeranz, 1967). The greater degree of branching found in water soluble polysaccharides may account for the greater effect noted.

The hydrogen bonding capacity of polysaccharides has also been found to be important by Patil et al. (1975b). They found that when flour constituents were mixed, there aggregation of pentosans, proteins, and/or glycoproteins. The pentosans and glycoproteins believed to act as a bridge between protein and carbohydrate components. Oxidizing agents may increase This interaction between starch and this interaction. pentosans was also found by other workers who noted that pentosans retard starch retrogradation (Kim and D'Appolonia, 1977).

Studies with cookies have shown that tailings decrease cookie spread. This has been related to a decrease in free water (Sollars, 1959; Sollars and Bowie, 1966).

Fiber Incorporation into Food Systems

Dough Systems

Pomeranz et al. (1977) incorporated cellulose products, wheat bran, and oat hulls into bread. Cellulose and bran increased water absorption while oat hulls decreased absorption slightly. They found that 5-7% cellulose or bran could be incorporated into bread although the fine structure was destroyed as seen through the electron microscope. Oat hulls imparted a gritty taste which was unacceptable. Other studies have

incorporated 16% bran into bread and muffins (Pyler, 1973).

Sugar snap cookies were formulated to contain 30% of the flour substituted with bran without affecting top grain, although spread was reduced, and 50% bran was found to be acceptable in an oatmeal cookie (Vratanina, 1978).

Batter Systems

One of the first studies on cakes was conducted by Brockmole and Zabik (1976) to evaluate the feasibility of incorporating wheat bran and middlings into white layer cakes. Substitution levels included 4, 8, and 16% bran, 12% middlings, and 16% bran plus 12% middlings to approximate a whole wheat flour. These substitutions were based on the weight of the flour. Both 60 and 70% flour extractions were used.

Substitution of bran and/or middlings resulted in increased tenderness, while parameters such as volume, pH, moisture, symmetry, uniformity, and shrinkage were not affected. Sensory scores indicated that all of the cakes were acceptable. Since a red wheat was used for this study, analyses with the Hunter Color Difference Meter showed that the cakes were darker with higher yellow and red coloration.

To minimize the effects of bran upon flavor and color, a study was conducted by Rajchel et al. (1975) in which bran and middlings were added to banana, cocolate,

nut, and spice cakes. The only adverse effects noted for volume accompanied the addition of bran or middlings to banana cakes, and middlings or the combination of bran and middlings to chocolate cakes.

Since these cakes contained a greater percentage of sugar based on the weight of the flour, the volume decrease that was noted probably resulted from an imbalance in moisture. Most of the sensory parameters measured (color, moisture, tenderness, texture, flavor) were unaffected by fiber addition. The general acceptability of all of the cakes was good, indicating that bran and middlings could be used in levels up to 16% bran plus 12% middlings in layer cakes to increase the level of fiber in the diet.

A third study was initiated to see how high a level of bran could be used before the effects were fairly detrimental. White cakes were made using flour which had been substituted with 30,50, and 70% bran. A soft red wheat bran was again used for this study (Springsteen et al., 1977).

Batter viscosity increased significantly with the addition of bran. This was believed to be due to the high molecular weight polymers such as cellulose and hemicellulose. Cake volume decreased with increasing levels of bran substitution, but was not significant until 50% of the flour was replaced by bran.

In general only the cakes with 70% of the flour substituted with bran exhibited lower sensory scores. Flavor was one exception. This was partly due to the age of the bran. The bran had been stored at room temperature six months prior to being used, and rancidity development was evident. This was thought to be caused primarily by the lipoxidase content of the bran, which catalyses oxidative rancidity. When the bran was ground, some bran particles were more resistant to grinding than others. Cakes made with bran of different particle sizes were compared. Cakes made from the smaller particle size (297μ) the greatest increase in viscosity, however, volume was not altered. Cakes containing bran of larger particle size (420µ) scored lower for most sensory characteristics. Regrinding the bran improved scores cell size, grain, softness, as well as total score, indicating that particle size affects cake quality.

EXPERIMENTAL

Cake Baking Studies

Material Procurement

Wheat brans of the following varieties were obtained from the Soft Wheat Quality Laboratory in Wooster, Ohio: Ionia and Yorkstar (soft white wheats, (SSW)); Commanche and Shawnee (hard red wheat (HRW)); Oasis and Arthur (soft red wheats (SRW)). In addition, a commercial wheat bran and oat bran were donated by Quaker Oats, soy hulls by General Mills, and a corn bran by Lauhoff Grain Company. Mechanically ground cellulose was obtained from the Berlin-Gorham division of Brown Company. Other cellulose types were supplied by the Avicel Department of the FMC Corporation. Descriptions of the cellulose types are as follows:

Type A - Solka-Floc. BW-200 Pharmaceutical. White to cream-colored, free flowing powder containing no obvious foreign material; screen analysis, < 0.5% on 35 mesh, not < 90% through 100 mesh, not < 75% through 200 mesh. Average particle size, 30-35μ.

Type B - Avicel PH-101. Average particle size, 50µ;

- particle size specification, +60 mesh < 1.0% and +200 mesh < 30%.
- Type C Prototype sample #170-2. Average particle size, $5-20\mu$, without hydrogen bonded aggregate, as in Avicel PH-101.
- Type D Prototype sample #174-2. Average particle size, $150-225\mu$, floc-like, particle size larger than Avicel PH-101.
- Type C+CMC Prototype sample #170-1. Average particle size, $5-20\mu$, same as sample #170-2, with addition of 2% CMC to serve as a protective colloid and to aid in dispersion of the hydrolyzed cellulose.
- Type D+CMC Prototype sample #174-1. Average particle size, 10-225µ, same as sample #174-2, with addition of 2% CMC during processing.
- 85% Type D/15%P Prototype #174-2. 85% sample #174-2 coated with 15% NF grade citrus pectin, medium viscosity (P).
- 70% Type D/30%P Prototype #174-2. 70% sample #174-2 coated with 30% NF grade citrus pectin, medium viscosity.

The pectin-coated prototypes were prepared by cospray drying slurries of cellulose and pectin. Straight grade soft red wheat flour (72% extraction) was obtained from Mennel Milling Company. Common lots of sugar, salt, shortening, nonfat dry milk, and baking powder were

obtained from Michigan State University Food Stores. The baking powder was specially ordered to insure freshness.

Dried egg whites were supplied by Seymour Foods, Inc.

Method of Preparation

Flour Preparation. Where necessary, the brans which had been frozen at 1.5-2.0°C were ground to a particle size similar to that of the control flour to eliminate mouthfeel differences. This was done using a Udy Cyclone Sample Mill, model MS. The bran remaining on the top 2 sieves (U.S. standard sieves 30 and 40) were reground until the bran passed through them. Sizing was done using a Roto-Tap Testing Sieve Shaker, model 4589 (Donelson and Yamazaki, 1972). The percent of the bran held on each sieve was recorded as well as the number of sievings necessary to reduce the bran.

Both the control flour and individual fiber sources were mixed for 30 minutes in a Liquid-Solids Blender, model S44EXAK-989 to insure uniform particle distribution.

Cake Mix Preparation. Layer cakes were prepared according to the standard AACC formula method 10-90 shown in Table 5. Prior to preparation of the cakes, a common mix was prepared containing all ingredients except water, 30% of the flour and the baking powder. The preweighed ingredients were mixed in a Food Mixer, model 100A to insure uniform distribution and then subdivided into

Table 5. Formula for standard high-ratio white layer cakes

	Amount		
Ingredient	g	% (flour basis)	
Flour	150.0	100.0	
Sugar	210.0	140.0	
Shortening	75.0	50.0	
Non-fat Dry milk	18.0	12.0	
Dried egg whites	13.5	9.0	
Salt	4.5	3.0	
Water	232.5	155.0	
Baking powder ¹	7.87 - 8.25	5.5 - 5.7	

Added according to schedule based on barometric pressure as outlined in AACC method 10-90.

appropriate amounts for one replication, sealed in polyethylene bags, randomly numbered, and held at 1.5-2.0° C until used. The fiber sources which were to be added were also weighed and sealed in bags, randomly numbered and held at 1.5-2.0°C until used.

Baking order of the five replications of the control and eight variables was also completely randomized. Five cakes were prepared on one baking day. The premix was warmed to 20°C before being blended in a Kitchen Aid Mixer Model K5-A for 1 min at low speed (145 rpm) with the flour or cellulose and the amount of baking powder prescribed for the barometric pressure of the day using the AACC Method 10-90 (AACC, 1962). Sixty percent of the water was added, and the mixture was allowed to soak for 2 min and subsequently mixed 1/2 min at low speed (145 rpm) for 4 min. The remaining water was added in two equal portions, in a similar mixing pattern, with mixing times of 2 min at medium speed after each water addition.

Baking Procedure

Four hundred twenty-five (425) grams of batter were scaled into greased and lined 8 inch round cake pans. The cakes were then placed on the middle shelf of an Etco forced convection oven, model 186A equipped with a Honeywell versatronic controller, and baked for 25 min at $177 \pm 2^{\circ}$ C. After removal from the oven, the cakes were allowed to cool 30 min in the pan. They were then removed

from the pan and allowed to cool 30 min on racks before cutting.

Preparation of Samples

After obtaining volume measurements, the layer cakes were cut for evaluation (Figure 8). A specially manufactured cutter was used to cut samples for tenderness evaluation. All samples were wrapped in Raynolon Food Service Film to prevent dehydration. The sample used for color analysis was frozen at 1.5-2.0° C. Moisture, tenderness, and sensory evaluation were determined the same day.

Objective Measurements

Objective measurements run on the cakes included tenderness, crumb and crust color for cakes containing bran, crumb color for the cakes containing cellulose, volume and moisture. In addition viscosity of the batter was determined.

Batter Viscosity. Batter viscosity was determined by a Brookfield Viscometer, model RVF-100, using a no. 7 spindle rotating at 10 rpm. The reading was taken after the dial had made one complete revolution and the reading was then multiplied by the appropriate conversion factor to express viscosity in poise.

<u>Volume</u>. Cake volume was determined using the AACC method

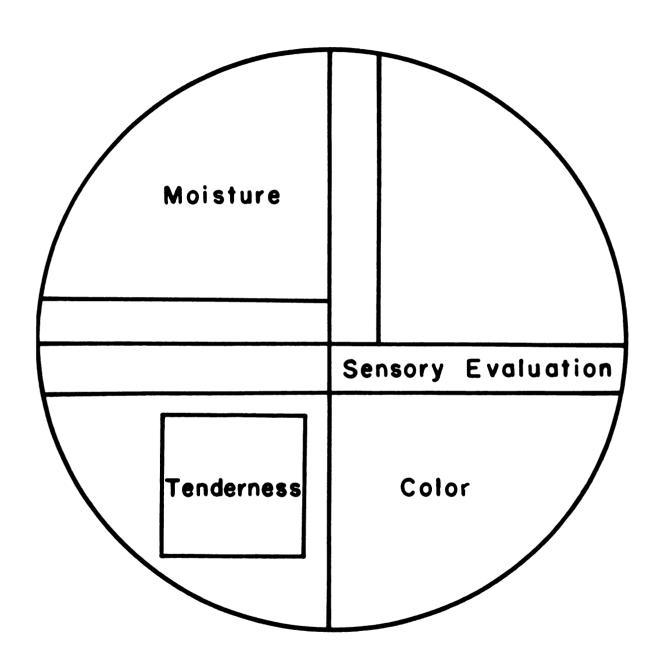


Figure 8. Cutting diagram of the cakes.

10-91 to obtain shrinkage, volume, symmetry, and uniformity indices (AACC, 1962).

<u>Color</u>. The Hunter Color Difference Meter, model D25, was used for color determination. The instrument was standardized against a white tile (L=93.0, a_L =-0.6, b_L =-0.1) for the cakes containing cellulose, and a yellow tile (L=82.5, a_L =-3.5, b_L =26.2) for the cakes containing bran. The cakes were cut in half horizontally. Crumb color was determined on all cakes. In addition, crust color was determined on the cakes containing bran. Two readings of each measurement were made.

Tenderness. Tenderness values were determined using the standard shear compression cell of the Allo-Kramer Shear Press, model SP12, equipped with an electronic recorder, model E2EZ using the procedure outlined by Funk et al. (1965). Cake samples 5.73 cm square were weighed to the nearest 0.1 g and placed in the cell. The tenderness value was expressed as pounds force per gram according to the formula,

Tenderness = $\frac{\text{Reading} \times \text{Ring} \times \text{Range}}{\text{Sample wt.} \times 100 \times 100}$

Moisture. Cake moisture was determined using the AACC procedure 44-40 (AACC, 1962). Approximately 2 g samples,

weighed to the nearest .0001 g, were dried for 5 hrs. at 90°C under a vacuum of 27 inches of Hg in a Hotpack vacuum oven, model 633. Samples were reweighed after cooling in a desiccator. Percentage moisture was calculated according to the formula,

% Moisture =
$$\frac{\text{wt. of moisture lost (g)}}{\text{sample wt. (g)}} \times 100$$

Subjective Evaluation

Training sessions held prior to taste panel evaluation acquainted the panel members with the score card and characteristics of the cakes which would be judged. The layer cakes were then evaluated by a panel of five trained judges for internal characteristics following a modification of the method outlined in the AACC method 10-90 (AACC, 1962). A sample score card appears in the Appendix.

Analyses of Data

The data were analyzed for variance and the following orthogonal comparisons calculated using the procedures described by Snedecor and Cochran (1967): 1) for the cakes containing cellulose - control cakes versus cakes containing Type A and Type B cellulose; cakes with Type A cellulose versus cakes with Type B cellulose; cakes with Type B cellulose; cakes with

size (150-225μ versus 5-20μ); pectin versus no pectin coating; and percentage of pectin coating. 2) for the cakes containing bran - control versus cakes containing bran; cakes made with wheat bran versus cakes with non-wheat bran; corn versus oat, versus soy brans; commercial bran versus other wheat bran; Commanche versus Shawnee; Oasis versus Arthur; Ionia versus Yorkstar. In addition, the following two nonorthogonal comparisons were made: control versus cakes containing wheat bran; control versus cakes containing non-wheat bran.

Analyses of Fiber Sources

The wheat brans, corn, soy, oat bran and control flour were analysed for moisture, ash, protein, crude lipid and fiber content. Moisture was analysed using the AACC procedure 44-40 (AACC, 1962) as previously described.

Ash

Approximately 0.5 g samples were weighed to 0.0001 g, ashed in a Barber-Colman Oven, model 293C at 525°C for 48 hours, cooled in a dessicator and then reweighed. Percentage ash was determined according to the formula

Crude Fat

Samples which had been previously dried were weighed to 0.0001 g, wrapped in filter paper and extracted in a soxhlet apparatus for six hours. The ether extracts were filtered through medium porosity glass fritted filters. The ether was then evaporated off over a steam bath and the fat flask dried at 100°C for 30 minutes. Percentage fat was calculated as:

% Fat = wt. of flask after extraction - wt. of flask sample wt.

Protein

Protein analysis was performed on the wheat samples. Total nitrogen was determined using the Kjeldahl method described by McKensie (1970) with slight variations. Approximately 30 mg of material was digested with 1.5 g of powdered potassium sulfate, 2.0 ml of sulfuric acid and .5 ml of mercuric sulfate soluion.

The mercuric sulfate solution was prepared bν dissolving 13.7 g of mercuric sulfate in a total volume of 100 ml of 2 M sulfuric acid. After digestion the flasks were cooled and diluted with deionized water. The flasks were transferred to a micro-Kjehdahl distillation As the water began to distill into the flask, 15 ml of sodium hydroxide - sodium thiosulfate solution were added and the mixture steam distilled into 10 ml of boric acid 50 ml beaker. indicator solution in a The sodium hydroxide - sodium thiosulfate solution was prepared by dissolving 200 g of sodium hydroxide and 12.5 g sodium thiosulfate in 400 ml of water.

The boric acid indicator solution was prepared by adding 6.67 mg methylene blue dissolved in 100 ml of water and 13.3 mg methyl red dissolved in 10 ml ethyl alcohol, combined with enough saturated boric acid to bring the volume up to one liter.

Distillation was continued until 40 ml had been collected. The beaker was lowered and the condenser tip

rinsed with deionized water. A few more ml were collected and then distillation was halted.

The distillate was titrated back to a purple end point with 0.0905 N hydrochloric acid. The hydrochloric acid had been standardized with Tham using p-sulfo-o-methoxybenzene azo dimethyl-1-naphthylamine indicator. Percentage protein was calculated as follows:

% Protein =

(sample - blank)(Normality HCl) × meq wt N × 5.8 × 100 sample wt.

Fiber Separation

Lipid Extraction. Samples weighing aproximately 0.2 g were weighed to 0.0001 g in 50 ml stainless steel centrifuge tubes. Twenty five ml of diethyl ether were added and the material mixed on a test tube mixer, Scientific Products Model S8220. Extraction was to proceed approximately 20 hours at room temperature. The tubes were then centrifuged at 29,000 g for 15 minutes. The supernatant was poured off and filtered through 60 ml medium porosity glass fritted filters. ml of diethyl ether were added, the tubes mixed and reextracted for an additional 4 hours. The tubes were once more centrifuged at 29,000 g for 15 minutes and the supernatant poured off through the filters. The samples were air dried at room temperature until there was no more ether remaining.

Total Water Solubles. Twenty five ml of deionized water were added to the centrifuge tubes. They were then shaken on a Burrel shaker for 4 hours at room temperature. The tubes were centrifuged at 29,000 g for 15 minutes and the supernatant filtered through the same glass fritted filter into glass 50 ml centrifuge tubes. Five ml of water were added to the flour samples, mixed, recentrifuged and poured off. After filtration the filters were rinsed with a few ml of water. The collected filtrate was heated at 100° C for 30 minutes to assure inactivation of natural enzymes. A fraction of this material was diluted and frozen for later sugar analyses. Pentose and hexose content were measured.

Water Soluble Fiber Components. Nine ml of the total water solubles were pipetted into glass 50 ml centrfuge tubes. One ml of amyloglucoxidase (glucoamylase, 1,4-glucan glucohydrolase; E.C. No. 3.2.1.3, Sigma Chemical Co.) and 1.2 ml of 2 M acetate buffer pH 4.5 were added. The tubes were shaken on a Burrell shaker at room temperature for 18 hours. The amyloglucoxidase solution contained 1 mg enzyme per ml (Southgate 1976a).

After starch extraction the solution was dialysed in 0.39 in tubing against deionized water at room temperature for 24 hours to remove solubilized starch and water

soluble sugars. A fraction of the remaining solution was diluted and frozen for later sugar analyses. Pentoses, hexoses, and uronic acids were measured.

Water Insoluble Starch. Nine ml of water were used wash the small particles remaining on the glass fritted filters into the tubes containing the unextracted material. This was then heated at 100°C for 30 minutes to gelatinize the starch. After cooling, 1 ml amyloglucoxidase (1 mg per ml) and 1.2 ml of 2 M acetate buffer pH 4.5 were added. Several drops of toluene were added to prevent microbial growth, and the tubes were mixed and placed in an incubator at 37° C for 18 After 30 - 35 ml of 95% ethanol had been added, the tubes were centrifuged at 29,000 g for 15 minutes and filtered glass fritted gouch crucibles into through coarse volumetrics. The material was remixed with approximately 20 ml of ethanol, centrifuged and filtered. Approximately 5 ml of ether were added and mixed. The tubes were centrifuged for 5 minutes, and the supernatant again filtered. The volumetrics were made to volume, a portion diluted, and frozen for sugar analyses. Hexoses and pentoses were measured. The ether extracted material air dried, and the material remaining on the filter scraped and readded.

Protein Removal. Ten ml of water, 10 ml of 0.2 N

hydrochloric acid, and approximately 30 mg of pepsin (from hog stomach mucosa 2x's crystallized, E.C. No. 3.4.4.1 Sigma Chem Co.) weighed to 0.0001 g and a few drops of toluene were added and mixed. The mixture was incubated at 37°C for approximately 20 hours. Twenty ml of ethanol were added. Centrifugation, washing, and filtration were the same as described for starch. The same gouch filter was used for a given sample throughout the exraction procedure.

Pectin Extraction. The method used for pectin extraction was similar to that of McComb and McCready (McCready and McComb. 1952). Forty ml of 5% (wt/vol) EDTA were added and mixed to the extracted material to sequester divalent ions. The pH was then adjusted to 11.5 with 1 N NaOH demethylate the pectin molecules. After constant stirring for 30 minutes on a Corning Hot Plate Stirrer, model PC-351, the pH was adjusted to pH 5-5.5 with glacial acid. Approximately 30 acetic mg οf pectinase (polygalacturonase, poly- α -1,4-galacturonide glycanohydrolase from Aspergillus Niger E.C. No. 3.2.1.15 Sigma Chemical Co.) weighed to 0.0001 g were added and stirring continued for 1 hour. The entire mixture was poured onto the appropriate filter over a volumetric and each tube rinsed with enough water to transfer all of the material onto the filter. After filtration was complete, the filters were rinsed with water. The filters were removed

and rinsed with small portions of 95% ethanol and then anhydrous ethyl ether. This was allowed to air dry at room temperature, the volumetrics were made to volume and portions diluted, pipetted into test tubes and frozen for later sugar analyses. Hexoses, pentoses, and uronic acids were measured.

Water Insoluble Hemicelluloses. The material filter was scraped into 50 ml glass centrifuge tubes. ml of 5% hydrochloric acid (wt/vol) was used to rinse particles from the filter into the centrifuge tube. exception was the control flour which did not have enough material to transfer. The centrifuge tubes were then placed on a Kjeldahl digestion unit and heated for minutes at low setting. The tubes were constantly turned and mixed several times during the heating on the test tube mixer. The contents of the tubes were not allowed to The contents were then filtered boil. through appropriate filter into volumetrics. Hot 5**%** HCl was poured directly onto the filter containing the control filter was rinsed with HC1. flour. Each 5**%** The volumetrics after this step were removed and new volumetrics used for the second through fourth extraction. These extractions were carried out exactly as the After the last extraction, the filters were rinsed with water. The volumetrics were then made to volume, a portion diluted and pipetted into test tubes and frozen.

Hexose, pentose, and uronic acid content were measured.

The material remaining on the filters was rinsed with ethanol and then anhydrous ethyl ether and air dried.

Cellulose Extraction. A few drops of 95% ethanol added to the material on the filters. The filters were placed in 83 x 48 mm beakers, and approximately 25 ml cold 72% sulfuric acid (wt/vol) was added. A glass stirring rod was placed in each and the mixture stirred until the material dispersed, covered with parafilm and refrigerated at 1-2°C for 48 hours. Each day the mixture stirred and more acid added if needed to cover the was material. After 48 hours the acid remaining in the filter diluted with water (approximately 4:1) and filtered into volumetrics. The acid in tmhe beakers was also diluted and filtered. The material was rinsed with water, 95% ethanol and ethyl ether and air dried. More cold 72% sulfuric acid was added and reexracted overnight. Dilution and filtration into the same volumetric was performed. The beakers and filters were rinsed with water into the volumetrics. Volumetrics were made to volume, a portion diluted and pipeted into test tubes and frozen for later analyses. Hexose and pentose contents were measured.

<u>Lignin Quantitation</u>. The filters were rinsed with acetone and dried at 100°C under vacuum for 30 minutes. They were

then cooled in a dessicator and weighed with the stirring rods still in them. They were then asked at 525°C in a Barber-Colman Oven for 24 hours, cooled and reweighed. Percentage lignin was calculated as the difference in weight divided by sample weight times 100.

Sugar Quantitation

Hexoses. Hexoses were measured using the anthrone reaction (Hassid and Newfeld, 1964). Test tubes containing 1 ml solution were taken from the freezer and thawed. Five ml of anthrone solution were added and the solution mixed on the test tube mixer. The anthrone was made by dissolving 0.1 g anthrone in 76 ml sulfuric acid and 24 ml water. The solution was not kept longer then 36 hours.

The test tubes were heated at 100°C for 20 minutes, then cooled to room temperature in tap water. The concentration of hexose was determined by measuring transmission on a Beckman Spectrophotometer model DB-G at 620 nm. Glucose standards were run with each set of measurements. The glucose was dried over phosphorous pentoxide at room temperature. A 1% solution was made and frozen. Standards were diluted from this solution.

<u>Pentoses</u>. Pentoses were measured using the orcinol method (Albaum and Umbreit, 1947). Test tubes containing 3 ml of sugar solution (between 0 and 20 μ g/3 ml) were removed

from the freezer and warmed to room temperature. Three ml of ferric chloride solution and 0.3 ml orcinol solution were added and the mixture stirred. The ferric chloride solution was made by dissolving 0.1 g FeCl per 100 ml HCl. The orcinol solution was made by dissolving 5 g orcinol per 50 ml of 95% ethanol. The test tubes were heated at 100°C for 45 minutes, cooled to room temperature in a water bath, and the transmission read at both 670 and 600 nm on the Beckman Spectrophotometer. Xylose standards were run with each set of measurements. se standards were made from xylose which had been dried over phosphorous pentoxide. Standards were stored frozen at 1.5-2.0°C.

Uronic Acids. Uronic acid concentration was measured the carbazole reaction (Dische, 1947). Test tubes containing 1 ml of sugar (0 to 100 µg/ml) were taken from freezer and warmed to room temperature. Six ml of the concentrated sulfuric acid were added and mixed. The test tubes were heated at 100°C for 25 minutes and cooled to room temperaure in tap water. Two tenths ml of 0.1% carbazole soom temwere added and the test tubes mixed. After 2 hours the transmittance was read on the Beckman Spectrophotometer at 525 nm. Galacturonic acid standards were run with each set of measurements. The carbazole solution was made by dissolving 0.1 g carbazole in 100 ml of purified 95% ethanol. The ethanol was purified by refluxing 1 l of 95% reagent grade ethyl alcohol with 4 g

of zinc dust and 4 ml of 1:1 sulfuric acid for 24 hours. This was distilled in glass. Four g of zinc dust and potassium hydroxide were added and the alcohol redistilled in glass. Galacturonic acid standards were made from galacturonic monohydrate which had been dried over phosphorous pentoxide. A 1% slution was stored at 1.5-2.0° C. Standards were diluted from this stock solution.

Blanks and Recoveries. Blanks were run on the following extractions: starch, protein, pectin; hemicellulose. Recoveries were run on starch using purified corn starch; hemicellulose, using carrageenan and agar; cellulose, using microcrystalline cellulose (Avicel PH101). The starch was purified according to Schoch (1942). In this procedure, one part starch is refluxed with three parts 85% methanol for two hours and filtered. This is repeated five times.

Data Analyses

The data were analyzed with a PDP 11 computer. Standard curves for glucose, xylose, and uronic acids were run using a composite of all of the individual standard values measured. Concentration was plotted versus absorbance and a least square fit used to determine the slope and y intercepts.

Total micrograms of hexose and uronic acids were calculated by determining the micrograms in each test tube

((absorbance - y intercept)/slope) and multiplying by the appropriate dilution factor. Total micrograms in the blank were subtracted where appropriate.

Total micrograms due to pentoses were determined using simultaneous equations since hexoses absorb at the same wavelength. Absorbances were measured at 600 and 670 nm. The absorbtivity of glucose and xylose were determined at both wavelengths and the following equations solved:

ABS₆₇₀ - blank = CONC pent pent hex hex
$$(\epsilon_{670})$$
 + CONC (ϵ_{670})

ABS₆₀₀ - blank = CONC pent pent hex hex
$$(\epsilon_{600})$$
 + CONC (ϵ_{600})

The appropriate dilution factor was again used and total micrograms due to enzyme interference again subtracted out. The percentage of each substance was calculated by:

% =
$$\frac{\text{total } \mu g}{\text{sample wt. } \mu g} \times 100 \times 100$$

The factor was calculated to convert monosaccharide polysaccharide by using the molecular weight of molecules linked versus 2 separated units. Factors are as follows: hexose = .95; pentose = .94; uronic acids = .86. The factor used for uronic acids also took into account purity of the galacturonic acid standard. Since the standard was in the monohydrate form the factor 194/212 The purity checked by titration with NaOH was used. sample was only 98.6% that the pure. percentage of uronic acid and hexose to pentose were also calculated for each extraction.

Analyses of Data and Prediction Equations

The data from each extraction and each sugar measured were analysed for variance and the same orthogonal comparisons made as were made for the cake series using brans. In addition the following data were combined and analysed as total water insoluble hemicellulose: pentoses from the starch, protein, pectin, cellulose, and water insoluble hemicellulose fractions plus the uronic acids and hexoses from the two water insoluble hemicellulose fractions.

Statistical programs LS and LSSTEP were run on the CDC 6500 to obtain prediction equations for cake parameters using fiber components as the independent variables.

RESULTS AND DISCUSSION

Discussion of the Fiber Methods

The scheme used in these analyses is shown in Figure Southgate's scheme is shown in Figure 10 (Southgate, 1969). The two schemes differ in a variety of ways. Southgate's separation procedure extracted water solubles with hot water which can cause the degradation and solubilization of pectin and hemicellulose which are not soluble at room temperature (Alberscheim et al., 1960; Whistler, 1965). Hot water has been found to decrease the lignin of woods (Sarkanen and Ludwig, 1971). Also all of the baking studies incorporating water soluble hemicellulose have been performed on materials extracted room temperature (Donelson and Wilson, 1960a). After starch digestion, Southgate used ethanol to precipitate soluble hemicelluloses and the water remove the solubilized starch. The quantity of water soluble material was so small that this was not found to be feasible (Table 5). When dialysis was used to remove the solubilized starch, the water soluble values were much higher. Other researchers have used this method in the analysis of water soluble hemicelluloses (Kundig et al.,

1961; Tao and Pomeranz, 1967; Wrench, 1965).

A protein extraction step was added after the starch extraction since it has been found that if this is not done the proteins may condense with lignin, giving higher lignin values (Brauns, 1952).

I have also added a method for the removal analysis of pectin. Southgate's method removed pectin hemicellulose does with the which not allow differentiation between uronic acids which are part of the pectin versus the uronic acids which occur in the side chains of hemicellulose. Pectin recoveries were 90% on a dry weight and 85% on a wet weight basis, however, this did not include the methoxyl content. Methoxyl content of commercial samples on a wet weight basis is approximately 10% (Whistler, 1965).

Southgate boiled the material with 5% H₂SO₁₁ for hours to remove insoluble hemicelluloses. Tests on agar and carrageenan showed extensive degradation and formation under these conditions. products οf condensation Hydrolytic losses can occur with acid. Sugars in furanose form are most susceptible. No conditions yield complete hydrolysis with no degradative losses (Southgate, Heating 15 minutes gave optimum results, however, under similar conditions, the hemicelluloses of bran completely extracted. Multiple extractions therefore carried out. Four extractions were chosen,

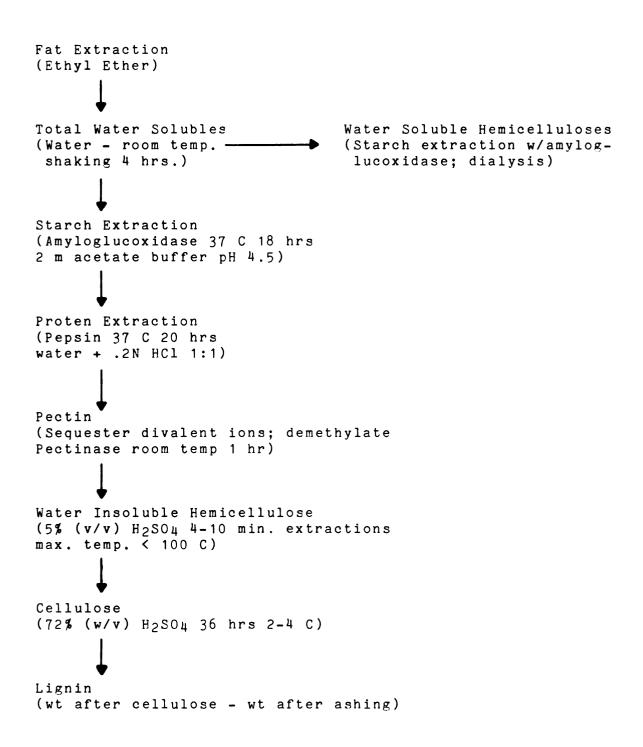
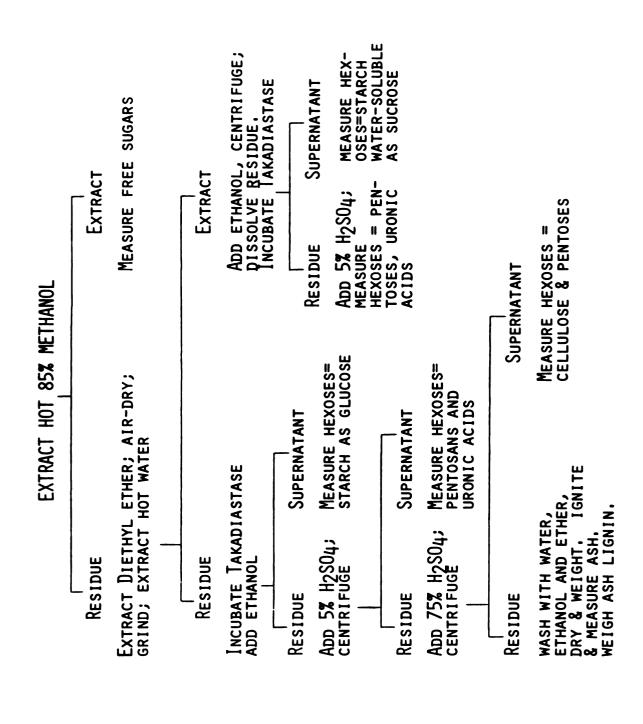


Figure 9. The scheme of analysis used for this research.

Figure 10. Southgate's scheme for the separation of fiber components.



since further heating extracted only a minimal amount of additional material. The material was not allowed to boil, since boiling reduced the level of recovered hemicelluloses (Table 6).

Cellulose recoveries were good using cold 72% sulfuric acid as Southgate suggested, however, the time was extended to 72 hours (Table 6). This was found to be particularly important when cellulose was extracted from soy bran which was extremely high in cellulose content.

Critique of Extraction Steps

<u>Fat Extraction</u>. The extraction of fat was performed on samples which were not dried prior to ether extraction. This was necessary since drying causes shrinkage of the material and later difficulties with solvent extraction. However, since drying was not performed, some water soluble material was extracted along with the fat.

Total Water Solubles. Total water solubles of various brans are found in the appendix. This extraction step includes water soluble sugars, starch, and hemicellular material.

<u>Water Soluble Fiber Components</u>. Individual values are found in the appendix. This extraction step includes water soluble hemicellular and pectic materials. These two could not be differentiated. The percentage of hexose

Table 6. Results of tests conducted on the methods

Component Measured	Material	Method	%
Water Soluble Pentoses	Bran	Southgate	.113
	Bran	Fractionation	.40
Water Insoluble Hexose	Agar ^{1,2}	Fractionation Southgate	99.2 condesation products
	Carragehnan 1,2	Fractionation	103.9
Pentose	Pran	Boiled 1.5 hour Fractionation fraction 1-3 4 5 6 cellulose	
Pectin	low methoxy citrus pectin ¹	Fractionation	90.04
Cellulose	Avicel pH 101 ¹	Southgate	98.9
Starch	Corn starch 1	Southgate	98.5

¹Based on ash and moisture free basis

²Corrected for oxygen in sulfate groups

 $^{^{3}\}mathrm{Based}$ on limited samples since results were low

⁴ Methoxyl content not included

measured was not included in the data since it could not be determined what portion remained from the soluble starch and sugars after dialysis. The uronic acid portion of this extraction was as high as the pentose portion. Since most of the materials measured have been found to contain little water soluble pectin, this may indicate that the water soluble hemicelluloses are higher than the water insoluble hemicelluloses in percentage uronic acids.

Starch and Protein. Values obtained from extractions are contained in the appendix. Both of these extractions contained pentoses, indicating that the enzymes extracted some of the hemicellular materials. This has also been found by other workers (Williams and 1935; Lintas and D'Appolonia, 1972). The analysed pentoses were added the to hemicellulose extraction. Hexoses from the protein extraction were not added since it was not clear how much contamination resulted from unextracted starch. The percentage of hexoses to pentoses was higher in this extraction than was found in the latter hemicellulose fractions. Individual sugar analysis of the hexoses would be of some help since hemicellulose fractions contain more galactose than most It was not found necessary in either extraction to subtract off blanks since the enzymes were relatively free of contamination.

Pectin. Individual pectin values are shown in the

This step had many inherent difficulties. appendix. of the major difficulties was that the enzyme found to be impure, containing large amounts of hexoses, pentoses, and uronic acids. Blanks were run in an effort subtract the interference. However, the quantity of uronic acids due to the enzyme was so much larger than the quantity in most of the brans (which are minimal in pectin content) that a small variance in the transmittance reading caused a large difference in the calculated value. Pentoses were also extracted by the pectic enzymes which hydrolyse 4-0-methyl-D-glucurono-Dhave been found to xylans, and many enzymes degrade L-arabino-D-xylans (Pigman and Horton 1970). Therefore some of the uronic acids measured as pectin were probably associated with the hemicellulose fraction extracted. Problems associated with the hexose fraction measured are the same as previously discussed for the protein extraction.

fraction Water Insoluble Hemicelluloses. This was measured in two parts (appendix) because it was believed that any material not already extracted would be extracted first portion leaving the second free of contamination. A better analysis of the minor components then be achieved. It appears that both fractions could were relatively free from contamination. Percentage of to pentose content was relatively components consistent among the wheat bran samples in a

The two extractions differed in the relative extraction. The fraction of proportion of the minor constituents. bran most easily hydrolysed contained less uronic wheat The method used for extracting acids and hexoses. hemicellulose was in most cases satisfactory. However. the control flour which was treated differently due to the small quantity of material was in most cases not extracted in this step.

Cellulose. The cellulose extraction (appendix) contained some pentose material which was not extracted with the hemicellulose material. This represents a fraction which is very resistant to extraction since further extraction of the hemicelluloses released very little additional Hemicelluloses are believed to be bonded to material. pentoses found cellulose and lignin, and the in bе fraction are believed to associated with a ligno cellulose fraction (Southgate, 1976b). It has estimated that up to 1-2% of the hemicellular monomer bonds are bonded to lignin (Sarkanen and Ludwig 1971). Of the wheat brans approximately 2.5% was not extracted. hemicellulose portion of the control which had not previously extracted was measured in this extraction. Therefore hexose values which were considered cellulose are probably high.

Standard Curves

Pentose. Figure 11 shows the standard curve of pentoses along with the interference due to hexoses and uronic acids. It has been suggested that the interference due to hexoses be eliminated by subtracting the absorbance at 600 nm from that at 670 nm (Whistler, 1962). This overcompensates since hexoses show a stronger absorbance at 600 nm. Simultaneous equations were therefore used to compensate for the interference. This could not be done with the uronic acids since they show the same absorption maxima. If the uronic acid determination had been more accurate, the quantity of uronic acids could have been subtracted since the absorbance due to them could have been calculated.

Hexose. The hexose measurement (Figure 12) gave fairly accurate results and had few problems associated with it. Interference from the other sugars was minimal. A composite standard curve was used since it was found that the curves varied around the actual value from day to day. This was established from the recovery data. Samples did not vary as much as did the standard curves.

<u>Uronic Acids</u>. Uronic acid standard curves (Figure 13) showed a large variance from day to day while the sample measurements varied to a lesser extent. Although a composite curve was used, standard deviations were very high. This method should be altered to obtain more

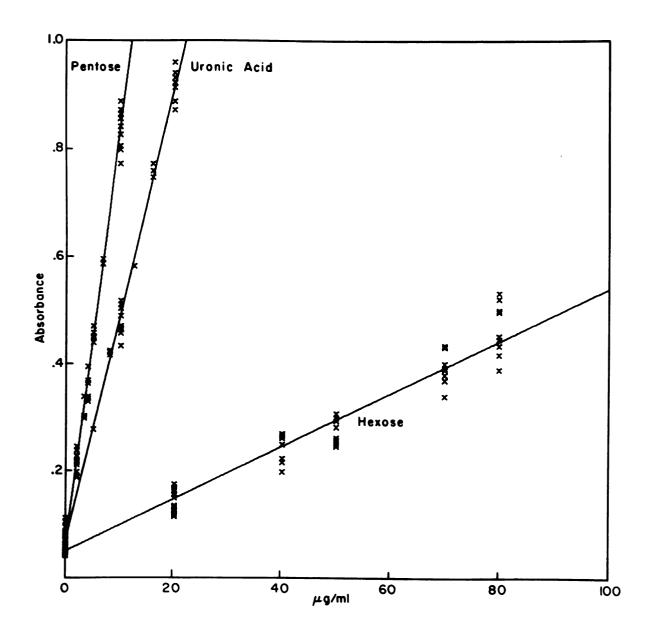


Figure 11. Absorbance curves at 670 nm of pentose, hexose, and uronic acids.

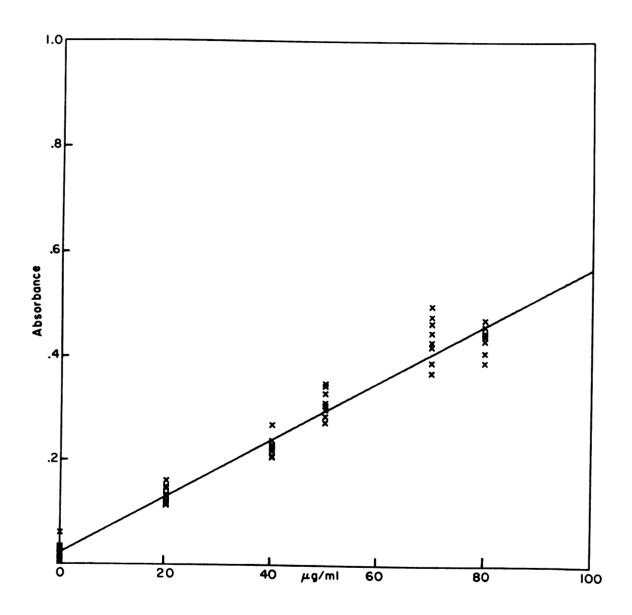


Figure 12. The absorbance curve for hexoses at 620 nm using the anthrone procedure.

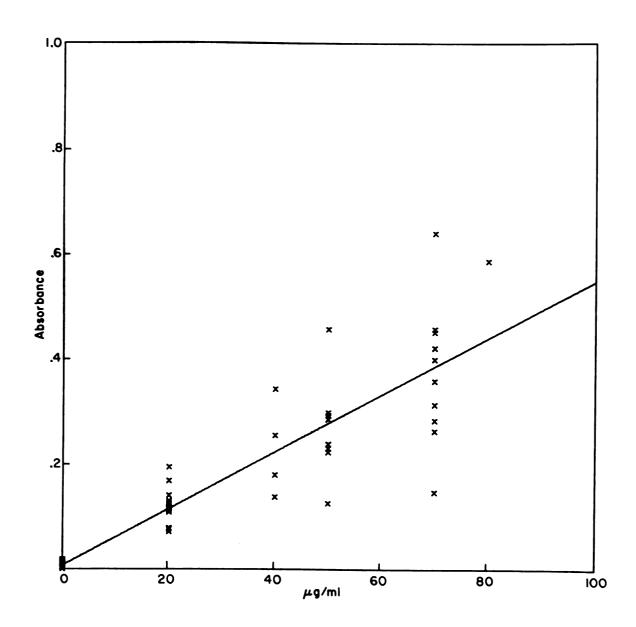


Figure 13. Absorbance of uronic acids using the carbazole reaction.

consistant results. It may be that fresh standards made on the day of analysis would have alleviated the problem. It has also been found that the color developed by this reaction depends on the nature of the neighboring residues. Calibration against some other method has been suggested (Southgate, 1976b).

Cake Results

Cake Containing Bran

Means and standard deviations of the objective and sensory evaluations of the cakes are shown in Tables 7;8 and 9;10 respectively. Statistical analyses of these data are presented in Tables 11;12 and 13 respectively. Pictures of these cakes are in Figures 14 and 15.

Control versus Other Cakes. Statistical analyses of the objective measurements of the control cakes versus those of the other cakes showed significant differences in most parameters including tenderness, viscosity and all of the color measurements (Tables 11;12). All of the batters containing bran were more viscous than the control batter (Tables 7;8). Color measurements of the crumb indicated that the cakes containing bran were darker and more yellow than control cakes. While the crumb of the control cake showed a green coloration, the cakes containing corn, oat, and soy bran were significantly less green, and the cakes

Objective measurements 1 of cakes prepared with 30% of the flour substituted with various cereal brans 7 Table

			Wheat	Brans		
	Commanche	Shawnee	Oasis	Arthur	Ionia	Yorkstar
Viscosity (poise)	453.0 ±40.4	423.0 +34.0	400.0 ±21.8	389.0	413.0 ±19.2	396.0
Tenderness (1b/g)	1.17±.11	1.19±.05	1.24±.07	1.15±.04	1.20±.06	1.3±.07
Volume (cm)	9.98±.26	10.10±.08	9.97±.30	9.92±.31	10.14±.42	10.24±.33
Moisture (%)	29.46±.30	30.00 . 35	29.59±.80	30.06±.22	29.71±.93	29.86±.24
Internal Color ²						
J	62.36±.23	61.96±.80	61.90±.43	61.66±.68	74.204.48	73.88±.63
a L	4.3±.56	5.16±.15	4.74±.51	5.12±.23	1.08±.38	1.50±.35
b _L	16.64±.18	16.50±.00	16.52±.19	16.98±.11	18.96±.15	19.28±.13
External Color ²						
-1	35.88±1.72	37.28±.99	37.52±2.30	35.50±2.86	39.08±3.81	39.50±1.67
П в	11.60±.95	12.40±.77	11.52±.79	11.54±.86	13.08±1.10	12.94±.77
$^{ m P}_{ m C}$	15.36±1.20	16.60±.42	16.20±1.09	15.20±1.95	17.94±2.76	18.16±1.33
				4. 5. 5. 5.		

5 replications Mean and stanard error of the mean based on

= yellowness

=redness, b

=greenness, +a

2Color values: L = lightess, -a

Objective measurements 1 of cakes prepared with 30% of the flour substituted with various cereal brans . ω Table

	Control	Corn	Soy	Oats	Commercial
Viscosity (poise)	75.0 ±23.2	581.0 ±60.2	652.0 ±109.5	577.0 ±66.0	378.0 ±30.7
Tenderness (lb/g)	1.49±.18	1.33±.08	1.38±.11	1.65±.06	1.13±.04
Volume (cm)	10.12±.30	10.96±.29	9.95±.20	9.75±.22	10.33±.19
Moisture (%)	29.14±.57	29.05±.12	28.32±.62	29.13±.43	29.86±.47
Internal Color ²					
IJ	84.26±.50	70.46±.59	68.16±4.00	73.74±.45	61.36±.95
a L	-2.60±.40	2033	02±.13	284.28	4.80±.22
P L	12.90±.10	22.70±.33	16.26±.18	16.54±.17	16.63±.22
External Color ²					
ı	46.12±4.65	43.54±1.92	41.08±3.75	43.72±5.52	36.78±2.92
a L	13.70±.74	13.14±1.19	7.32±1.49	12.38±1.40	11.33±.53
P L	22.14±2.69	20.90±1.07	17.18±1.31	20.26±2.51	15.33±2.10

5 replications. Mean and standard error of the mean based on

⁼ yellowness. م redness, 11 greenness, +a 11 lightness, -a 11 2 Color values: L

Sensory evaluations of cakes prepared with 30% of the flour substituted with various wheat brans Table 9.

			Variety of	Wheat Bran		
Sensory Characteristic	Commanche	Shawnee	Oasis	Arthur	Ionia	Yorkstar
Cells (30):						
Uniformity (10)	8.24±.36	8.32±.73	8.004.35	8.204.82	8.004.99	8.04±.77
Size of Cells (10)	9.16±.26	9.12±.69	7.92±.87	8.68±.66	8.404.58	8.204.58
Thickness of Walls (10)	9.16±.36	8.80 .32	8.84±.48	8.84±.59	8.48±1.01	8.04±.57
Grain (16):	13.12±.44	14.40±.69	14.00±.47	13.92±.59	14.40±.28	13.80±.62
Texture (34):						
Moistness (10)	9.20±.55	9.20#.66	9.32±.18	9.20≠.24	9.084.58	9.08±.33
Tenderness (14)	12.76±.43	13.40±.42	12.80±.49	13.24±.62	12.88±.84	13.12±.63
Softness (10)	9.20±.14	9.32±.33	9.204.58	9.24±.17	9.36±.33	9.28±.23
Crumb Color (10):	9.64±.36	60.4#.06	9.16±1.03	60.491.6	9.284.27	9.52±.18
Flavor (10):	8.92±1.00	9.52±.33	9.26±.76	9.52±.23	9.20±.32	9.20 .55
Total Points (100):	89.40±2.42	91.92±2.34	88.52±2.75	90.60±1.63	89.08±2.24	88.28±1.84

⁵ replications. o Mean and stanard error on the mean based

 2 Total possible points listed in parenthesis.

Sensory evaluations of cakes prepared with 30% of the flour substituted with various types of cereal brans and legume Table 10.

Sensory Characteristics ²	Control	Corn	Soy	Oats	Commercial
Cells (30):					
Uniformity (10)	8.80±.63	8.00±.40	6.32±.41	7.44±.26	8.44±.64
Size of Cells (10)	8.84±.50	8.64±.89	7.20±.66	7.04±.46	8.92±.88
Thickness of Walls (10)	8.28±1.01	8.68±.50	7.28±.50	6.36±.59	9.26±.30
Grain (16):	14.64±.52	12.16±.46	12.80±.71	11.84±.22	13.64±.88
Texture (34):					
Moistness (10)	9.36±.43	8.44±.83	8.52±.76	8.80±.68	97.08±.46
Tenderness (14)	12.28±.44	12.92±.52	12.96±.52	12.84±.61	13.56±.26
Softness (10)	9.16±.22	9.16±.46	9.32±.23	9.04±.26	9.36±.22
Crumb Color (10):	60.₹18.6	8.16±1.23	6.32±1.58	9.36±.38	9.64±.36
Flavor (10):	10.00±0.0	7.40±1.50	6.32±1.75	2.32±1.31	9.08±.41
Total Points (100):	91.20±2.43	83.56±3.96	77.04±5.11	75.44±2.41	91.00±2.16

5 replications. $^{1}{ t Mean}$ and standard error of the mean based on

 $^{^2}$ Total possible points listed in parenthesis.

F values of orthogonal comparisons for objective measurements of cakes containing various types of cereal brans and legume Table 11.

Comparison	Volume Indices	Tenderness	Viscosity	Moisture
Control vs rest	ηΟ.	28.91***	257.47***	2.26
Control vs wheat 1	90.	51.54**	178.26***	6.93**
Control vs non-wheat 1	.57	1.26	385.94**	1.29
Wheat vs non-wheat	2.57	94.54**	148.58**	35.74**
Corn vs Oat vs Soy	30.05***	18.85**	3.28*	7.37**
Commercial vs other wheats	2.14	2.51	1.86	1.
Hard vs soft wheat	41.	.50	3.64	. 15
Red vs white wheat	4.71*	.25	. 18	ηΟ.
Commanche vs Shawnee	.51	.62	.83	2.70
Oasis vs Arthur	60.	2.51	.11	2.04
Ionia vs Yorkstar	.51	. 13	.27	.22

Not orthogonal.

^{5%} level of probability.
1% level of probability. the Significant at Significant at

the 1%

the 0.1% level of probability. Significant at **

Table 12. F values of orthogonal comparisons for objective measurements of cakes containing various types of cereal bran

			Color) L		
Comparison		Interior			Exterior	
	ı L	๗	۵	ᆸ	๗	۵
Control vs rest	4118.64**	1032.17***	3491.00***	23.01***	17.75***	32.43**
Control vs wheat ¹	#**90.057h	1500.01***	2897.14**	33.38**	11.81##	***60° hh
Control vs non-wheat ¹	2062.85**	185.03***	3920.00***	4.16*	28.43**	**O†.8
Wheat vs non-wheat	946.91***	1386.67***	457.00###	30.66**	12.95***	29.57***
Corn vs Oat vs Soy	119.15***	.71	2208.33***	1.08	100.09**	6.10**
Commercial vs other wheats	278.79***	47.25***	105.67**	.20	3.17	1.48
Hard vs soft wheat	*** 16.799	145.83***	414.00***	1.16	64.	1.65
Red vs white wheat	2277.39**	552.08**	936.00**	3.84	10.95**	8.52**
Commanche vs Shawnee	1.21	15.42###	1.67	64.	1.60	1.19
Oasis vs Arthur	24.	3.00	17.67***	1.01	00.	.77
Ionia vs Yorkstar	61.	3.67	8.67**	40.	.05	ήΟ.

the 5% level of probability. the 1% level of probability. Significant at Significant at Not orthogonal
Significant
Significant

the 0.1% level of probability. *** Significant at

cakes F values of othogonal comparisons for sensory evaluations of containing various types of cereal bran Table 13.

		Cells			
Comparison	Size of cells	Thickness of walls	Uniformity	Crumb	Flavor
Control vs rest	2.69	.11	** † † 6	5.32	19.80***
Control vs wheat ¹	54.	2.90	4.35*	.73	2.93
Control vs non-wheat 1	12.49**	7.11*	23.00***	26.35***	95.53**
Wheat vs non-wheat	23.86**	50.36**	22.97***	52.82***	187.80**
Corn vs Oat vs Soy	*** 08.8	18.31**	9.38**	22.98***	84.26**
Cmmercial vs other wheats	1.13	3.97	1.03	. 10	. 19
Hard vs soft wheat	10.64**	3.30	.82	1.25	.05
Red vs white wheat	00.0	4.51*	80.	ήΟ.	ή2.
Commanche vs Shawnee	.01	.86	40.	.20	1.06
Oasis vs Arthur	3.26	00.	.26	1.76	L tr .
Ionia vs Yorkstar	.23	1.29	.01	.27	00.

Not orthogonal.

Significant at the 5% level of probability. Significant at the 1% level of probability.

^{***} Significant at the 0.1% level of probability.

Table 13 continued

		Tex	Texture		
Comparison	Grain	Moistness	Tenderness	Softness	Total pts.
Control vs rest	21.72***	2.00	**20.6	ιμ.	12.62***
Control vs wheat 1	7.59**	.55	10.10*	.61	1.03
Control vs non-wheat 1	***Ln.99	7.23*	4.95*	٠٥٠	73.39***
Wheat vs non-wheat	87.84 * *	11.35**	1.45	1.22	162.93***
Corn vs Oat vs Soy	3.78*	.58	70.	1.01	185.01**
Commercial vs other wheats	1.23	.13	4.01	ιη.	1.00
Hard vs soft wheat	1.54	.03	. 10	.01	1.97
Red vs white wheat	.31	.52	.01	.51	8 4.
Commanche vs Shawnee	12.90***	00.	3.43	.36	1.98
Oasis vs Arthur	90.	.13	1.62	ήΟ.	1.35
Ionia vs Yorkstar	2.83	00.	۲4.	. 16	.20

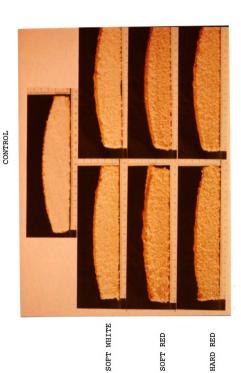


Figure 14. Pictures of the cakes containing the control flour and wheat brans.

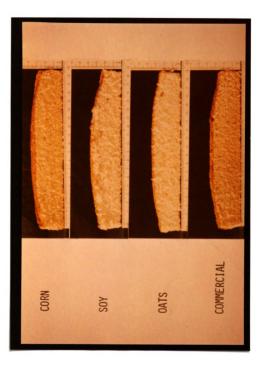


Figure 15. Pictures of cakes containing various brans.

containing wheat bran had a red coloration (Tables 7;8).

The crust of the control cake was lighter, more green and more yellow than that of all the other cakes.

Volume indices showed no significant difference between the control and the cakes containing any type of bran. Tenderness measurements, however, showed that the control cake was less tender than the cakes containing wheat bran. Moreover, moisture analyses indicated that the cakes containing wheat bran were more moist than the control cake. The moistness of cakes containing non-wheat bran, however, was not significantly different from the control cake.

Statistical analyses of sensory evaluations of the control cake versus cakes containing wheat bran showed very few significant differences (Table 13). The cakes with wheat bran did score significantly lower on measurements of uniformity of cells or grain. They were, however, significantly more tender than the control cakes.

The cakes containing non-wheat bran scored significantly lower on most sensory evaluations when compared with the control. These included cell size, thickness of cell walls, uniformity of cells, grain, moistness, crumb color, flavor, and total points. These cakes were more tender than control cakes, however.

Wheat versus Non-wheat Bran. Objective measurements of cakes containing wheat versus non-wheat bran showed

significant differences in all measurements but volume (Tables 11;12). Batters made from non-wheat bran were more viscous than batters containing wheat bran. The cakes made from non-wheat brans were less tender and less moist. Both the crust and crumb of cakes made from non-wheat brans were lighter than the crust or crumb of cakes containing wheat bran. While the crumb of cakes with wheat bran showed a red coloration, the crumb of cakes containing non-wheat brans had a slight green tinge.

Sensory scores indicated that cakes made from non-wheat bran were significantly less acceptable than cakes containing wheat bran for size of cells, thickness of cell walls, uniformity, grain, moistness, crumb color, flavor, and total points scored.

Non-wheat Brans. Although the cakes containing non-wheat brans as a whole scored significantly different from cakes containing wheat brans, there were also many significant differences seen among the cakes made with corn, oat or soy bran.

Objective measurements showed significant differences in volume, tenderness, viscosity, moistness and many of the color measurements (Tables 11;12). Although cakes made with soy and oats had similar volumes, the cakes made with corn bran had volumes greater than any other cake. The cakes made from oat bran were the least tender. The cakes made from soy bran were the least moist while the

		1
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		! !

batter was the most viscous. As expected, the crumb of cakes made from corn bran was more yellow than any other cake.

Sensory values showed significant differences in size of cells, thickness of cell walls, uniformity, grain, crumb color, flavor, and total sensory points (Table 13). The cakes made with soy bran scored lowest for crumb color, while cakes containing oat bran scored lowest for flavor and total points (Table 13).

Commercial versus Other Wheat Brans. There were no significant differences found among any of the sensory evaluations for the comparison of cakes containing commercial versus other wheat brans (Tables 11;12). The only significant differences in objective scores occurred in crumb color measurements (Table 12).

The crumb color was significantly darker, less red, and less yellow than the other cakes (Tables 7;8). However, when compared only to the cakes made with red wheat bran, the values for all color measurements were very similar, indicating that the commercial mix was primarily, if not totally, made from red wheat.

Hard versus Soft Wheat Brans. Statistical analysis of objective measurements of cakes containing hard versus those of cakes with soft wheat brans showed significant differences only in the crumb color (Table 12). The use

of soft wheat brans resulted in cakes which were lighter, less red and more yellow (Table 7). These differences were probably due to the white wheats.

The only difference in sensory scores that occurred among these cakes was in the size of the cells. The cakes made from hard wheat bran scored significantly higher for this measurement (Table 13).

Red versus White Wheat Bran. The comparison of objective measurements for cakes containing red versus white wheat bran showed the expected significant differences in crumb and crust color as well as significant differences in volume indices (Tables 11;12). The cakes containing white wheat bran were significantly higher in volume than cakes containing red wheat bran (Table 7).

Sensory scores indicated that the cakes made from white wheat bran had thicker cell walls than cakes containing red wheat bran (Table 13). No other sensory evaluations showed any significant differences.

<u>Differences Among Varieties</u>. Objective evaluations of cakes containing the wheat brans of different varieties but of the same class of wheat showed very few differences (Tables 11;12). The only significant differences which did occur were in some of the color measurements (Table 12). Sensory evaluations showed only one significant difference. This occurred when cakes containing Commanche

and Shawnee brans were compared. The cakes with Shawnee scored significantly higher for grain evaluation than did Commanche.

Cakes Containing Cellulose Derivatives

Means and standard deviations of the objective and sensory evaluations of the cakes are shown in Tables 14;15 and 16;17, respectively. Statistical analyses of the data are presented in Tables 18, 19, and 20. Pictures of these cakes are in Figures 16-18.

Control versus Types A and B. Mechanically ground (Type A) and hydrolyzed (Type B) celluloses were compared to the control. The only objective measurements which showed significant differences were batter viscosity and Hunter all values (Tables 18;19). Batters containing cellulose were more viscous than control batters as has been previously reported (Brys and Zabik, 1976). Hydrolyzing cellulose has been found to produce many fissures in the dry particles thereby contributing to the absorbent properties (Trauberman, 1961). However, comparisons of the mechanically ground versus hydrolyzed cellulose showed significant differences for batter viscosity and volume. Batter containing the ground material was more viscous and the resulting cake had an increased volume.

The control cake crumb had very low greenness values while the cakes containing cellulose had very low redness

Objective measurements 1 of cakes prepared with $30\mbox{\$}$ of the flour substituted with various types of cellulose Table 14.

Physical	Control		Cellulose	se Type	
Characteristic		А	В	Ĵ	D
Viscosity (poise)	144±166	486±268	236±182	230±108	263±91
Volume Index (cm)	10.6±0.2	11.5±0.3	10.0±0.3	9.9±0.1	10.5±0.5
Symmetry (cm)	1.1±0.2	1.1±0.3	0.5±0.4	η·ο∓η·ο	0.9±0.3
Uniformity (cm)	0.1±0.1	0.1±0.1	0.1±0.1	0.1±0.1	0.0+0.0
Shrinkage (cm)	1.1±0.2	1.1±0.4	1.2±0.2	1.2±0.2	1.32±0.2
Tenderness (lb/gm)	1.35±0.29	1.32±0.28	1.15±0.12	1.28±0.20	1.32±0.11
Color Values ²					
נו	83.9±1.5	84.8±2.3	83.9±1.3	84.6±1.5	85.8±0.8
e Li	-1.2±1.2	3.2±0.5	0.5±0.7	1.6±0.4	2.4±0.8
D L	12.4±0.5	12.1±0.2	11.9±0.2	12.3±0.1	12.0±0.4
Moisture (%)	28.8±0.5	28.4±0.4	28.1±0.6	27.1±1.1	28.1±0.4

5 replications ¹Mean and standard error of the mean based on

=yellowness. = redness; b =greenness; +a = lightness; -a ᆸ 2 Color values:

Objective measurements 1 of cakes prepared with 30% of the flour substituted with various types of cellulose. Table 15.

Characteristic		1		The second secon
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	٥	D	15% Pectin	30% Pectin
Viscosity (poise)	192±54	270±17	923±70	1970±245
Volume Index (cm) 9	9.9±0.1	10.3±0.3	9.8±0.1	8.5±0.6
Symmetry (cm) 0	0.5±0.1	0.2±0.2	0.6±0.1	0.3±0.7
Uniformity (cm) 0	0.0±0.1	0.1±0.1	0.1±0.1	0.3±0.1
Shrinkage (cm)	1.3±0.2	0.9±0.2	1.0±0.2	1.1±0.2
Tenderness (lb/gm)	1.29±0.24	1.36±0.15	0.99±0.11	0.88±0.39
Color values ²				
Т 85	5.7±0.9	84.7±2.8	85.7±1.3	83.8±1.3
0 Te	0.0±1.1	2.2±1.0	2.9±1.8	-0.6±1.6
b _L 12	2.0+0.3	11.7±0.5	12.1±0.4	13.1±0.7
Moisture (%) 28	8.2±0.3	28.0+0.8	28.4±0.5	27.4±0.7

 $^{1}{\rm Mean}$ and standard error of the mean based on 5 replications.

yellowness. 11 ۵ redness; 11 greenness; +a 11 = lightness; -a ᆸ 2color values:

Sensory evaluations 1 of cakes prepared with 30% of the flour substituted with various types of cellulose. Table 16.

Sensory	Control		Cellulose	se Type	
Characteristic ²		A	В	1	D
Cells (30):					
Uniformity (10)	9.1±0.7	8.2±0.2	7.3±0.5	7.8±1.3	7.5±0.9
Size of Cells (10)	8.4±0.2	8.8±0.4	7.9±0.4	8.1±1.0	8.1±0.5
Thickness of Walls (10)	8.0+0.2	4.0+6.8	7.4±0.2	7.7±1.2	7.8±0.6
Grain (16):	14.5±0.6	14.4±1.1	13.9±0.8	13.9±0.4	13.8±0.4
Texture (34):					
Moistness (10)	9.3±0.7	8.9±0.7	9.2±0.5	9.0±0.7	9.2±0.6
Tenderness (14)	11.5±0.6	13.7±0.1	13.2±0.4	12.7±0.2	13.3±0.3
Softness (10)	8.9±0.2	9.6±0.2	9.5±0.3	9.3±0.2	9.5±0.2
Crumb Color (10):	9.8±0.1	9.8±0.1	0.8±0.0	9.9±0.1	9.8±0.1
Flavor (10):	8.6±0.3	9.6±0.2	9.3±0.5	8.5±0.8	9.4±0.2
Total Points (100):	89.2±1.9	91.8±2.4	87.2±1.9	86.8±2.8	88.3±1.0

 $^1\mathrm{Mean}$ and standard error of the mean based on 5 replications.

 2 Total possible points listed in parenthesis.

Sensory evaluations 1 of cakes prepared with 30% of the flour substituted with various types of cellulose. Table 17.

Sensory ,	CMC w/Cellulose	llulose Type	1 1	Type D w
Characteristic ²	၁	Q	15% Pectin	30% Pectin
Cells (30):				
Uniformity (10)	8.1±0.8	8.1±0.4	6.8±1.2	2.2±0.4
Size of Cells (10)	8.5±0.6	8.6±0.3	6.4±1.2	3.6±0.5
Thickness of Walls (10)	8.3±0.4	η·0∓η·8	7.4±1.1	4.6±0.5
Grain (16):	14.1±0.9	14.7±1.0	14.6±1.2	13.1±1.0
Texture (34):				
Moistness (10)	9.7±0.2	9.4±0.5	6.5±0.4	5.4±0.8
Tenderness (14)	13.5±0.1	13.4±0.3	12.4±0.6	9.5±0.1
Softness (10)	9.5±0.3	9.6±0.2	9.0+0.6	6.0±0.7
Crumb Color (10):	0.8+0.0	0.0+8.6	0.8±0.0	9.3±0.9
Flavor (10):	8.8±0.6	9.7±0.2	9.0±0.3	7.3±1.0
Total Points (100):	90.3±1.4	91.8±1.7	80.4±5.0	61.1±3.0

Mean and standard error of the mean based on 5 replications.

 2 Total possible points listed in parenthesis.

8. Analyses of variance and orthogonal comparisons for data of cakes prepared with $30\,$ % of the flour substituted with various types of cellulose. 18. Table

				Mean S	Squares		
Source	d f	Viscosity	Volume Index	Symmetry	Shrinkage	Tenderness	Moisture
Total	† †						
Variables	80	17751.25***	3.20***	0.54**	0.07	0.29	1.19
Control vs Type A & B	-	1572.53*	40.0	42.0	00.0	60.0	1.12
Type A vs B	-	1567.50*	5.85**	##06·0	0.02	0.15	0.31
Type B vs C;D1	-	3.75	0.14	0.08	00.0	0.15	0.70
Particle Size	-	152.35	0.99	0.02	0.11	0.03	0.72
CMC vs none	-	12.17	0.08	0.53*	0.05	0.01	1.14
CMC + particle size	-	125.32	0.32	0.23	40.0	0.02	0.11
Pectin vs none	-	46712.75***	5.63**	0.65*	0.11	**16.0	0.14
% Pectin	-	27394.76***	3.97**	0.23	0.05	90.0	2.13
Within	36	227.65	0.43	0.10	0.05	0.12	44.0
-							

1Not orthogonal.

^{*} Significant at the 5% level of probability.
** Significant at the 1% level of probability.

^{***} Significant at the 0.1% level of probability.

Table 19. Analyses of variance and orthogonal comparisons for data of cakes prepared with 30% of the flour subsittuted with various types of cellulose

Source	d f	H	unter Color Va	alues
50u1 ee	<u> </u>	L	а	b
Variables	8	6.81	25.30**	1.62***
Control vs Type A & B	1	1.54	59.20***	0.84
Type A vs B	1	4.61	35.38**	0.36
Type B vs C;D ¹	1	11.53	14.50#	0.62
Particle Size	1	0.08	23.26**	0.90
CMC vs none	1	0.01	7.83	0.96
CMC + particle size	1	5.30	24.64**	0.54
Pectin vs none	1	7.07	10.75	2.05*
% Pectin	1	19.20	63.37***	5.30***
Within	36	6.02	2.77	0.33

¹Not orthogonal.

^{*} Significant at the 5% level of probability.

^{**} Significant at the 1% level of probability.

^{***} Significant at the 0.1% level of probability.

Table 20. Analyses of variance and orthogonal comparisons for sensory evaluations

	-		Mean	Squares		
			Cells			
Source	d f	Uniformity	Size of cells	Thickness of walls	Crumb Color	Flavor
Total	44					
Variables	8	20.08***	13.40***	7.56***	0.15	2.91***
Control vs Type A & B	1	6.17**	0.02	0.00	0.00	0.09
Type A vs B	1	1.94	1.94*	7.40***	0.00	0.14
Type B vs C;D ¹	1	0.48	0.16	1.12	0.01	0.43
Particle Size	1	0.13	0.05	0.07	0.00	3.87***
CMC vs none	1	0.97	1.06	2.05*	0.00	0.51
CMC + particle size	1	0.00	0.06	0.04	0.01	1.94
Pectin vs none	1	30.00***	31.62***	10.56***	0.26	5.13***
% Pectin	1	53.82***	19.60***	19.04***	0.58#	7.74***
Within	36	0.64	0.42	0.42	0.09	0.28

Not orthogonal.

Significant at the 5% level of probability.

^{**} Significant at the 1% level of probability.
*** Significant at the 0.1% level of probability.

Table 20 continued

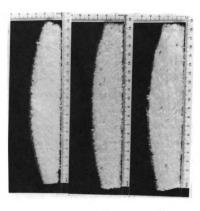
			Mean Squar	es	
		T	exture		
Source	Grain	Moistness	Tenderness	Softness	Total Points
Total					
Variables	1.27	10.96***	8.80***	6.58***	468.68***
Control vs Type A & B	0.48	0.23	12.29***	1.28***	1.23
Type A vs B	0.48	0.20	0.78*	0.04	52.90**
Type B vs C;D ¹	0.05	0.02	0.11	0.03	0.59
Particle Size	0.29	0.02	0.29	0.10	10.95
CMC vs none	1.57	1.25	0.97**	0.16	60.55**
CMC + particle size	0.90	0.20	0.04	0.02	5.48
Pectin vs none	0.01	33.71***	17.94***	13.07***	1030.19***
% pectin	5.48*	2.92**	20.74***	22.50***	933.16***
Within	0.76	0.34	0.13	0.12	6.75

values. Taste panelists, however, did not note any differences in acceptability of cake color (Table 20).

Sensory analysis of cakes with types A and B versus the control showed no significant differences in total sensory scores, however, the individual parameters of uniformity of cells, tenderness, and softness were significantly different (Table 20). The cakes containing both types of cellulose were found to have less uniform cell distribution, but were more tender and softer than the control. All of these cakes received excellent sensory scores.

Type A versus Type B. Comparisons of Type A versus Type B cellulose established significant differences in the following objective measurements: batter viscosity, volume, symmetry, and Hunter a values. Batter viscosity and volume have already been discussed. The cakes containing ground cellulose were more convex and more red. Total sensory scores for the cakes containing hydrolysed cellulose were significantly lower (Table 20), again indicating that hydrolysis had an adverse effect on cellulose functionality. However, since both cakes received good total scores, either could be substituted for flour at a 30% level to produce a high quality cake.

Type B versus types C and D. Cakes containing two prototypes of hydrolyzed cellulose (Types C and D) were



CONTROL

TYPE B CELLULOSE

TYPE A CELLULOSE

compared with the cakes containing PH-101 (Type B). Although it had been felt that changing the size of the cellulose aggregates might eliminate problems in mouthfeel when the products were used in low calorie formulations, few differences occurred in the data (Tables 18;19 and 20). All products functioned very well at this level of substitution in a high ratio cake formula.

Particle Size. The effect of particle size was determined by comparing cakes made from the two particle sizes, 5-20 μ and 150-225 μ , with and without 2% CMC. Few significant differences were noted (Tables 18;19;20). The only objective measurement that differed was in the a value, which indicated that the cakes made from the larger particle size were redder. Sensory scores differed in flavor. Although the cakes made from the smaller particle size received scores of 8.5 and 8.8 out of 10, panelists noted a slight chalky taste.

Carboxyl Methyl Cellulose Coated Cellulose. Coating hydrolyzed cellulose with CMC has been found to act as a protective coating and aid in the dispersion of the cellulose product (McCormick, 1970). Very few differences, however, were seen in the objective measurements of the cakes containing CMC coated cellulose (Table 18;19). However, sensory analyses indicated that CMC-coating did significantly affect total sensory scores

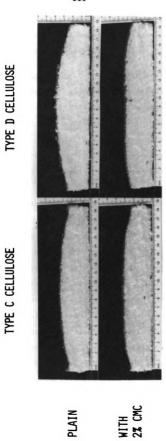


Figure 17. Pictures of cakes containing cellulose with and without CMC coating.

as well as cell wall thickness and tenderness (Table 20).

Cakes containing CMC-coated cellulose scored slightly

higher in all of these sensory attributes (Table 17).

Pectin coated Cellulose. Coating the hydrolyzed cellulose with pectin before its substitution for flour in cakes produced numerous significant differences in objective (Tables 18;19) and sensory (Table 20) data. Cake batter was extremely viscous and became more viscous with increasing percentage of pectin coating. The extremely high water-holding capacity of the percentages of pectins used resulted in an imbalance of the water in the high ratio cake formula causing decreased volume and flatter, less uniform cakes. Cakes with both levels of pectin coating required less force to shear than the cakes with cellulose of a similar particle size. Increasing the percentage of pectin had a significant adverse effect on batter viscosity, cake volume index, uniformity, Hunter aland blavalues and percentage moisture (Tables 18 and 19).

Analyses of sensory evaluations showed that cakes with pectin coated cellulose scored significantly lower for all characteristics except grain and crumb color (Table 20). Moreover, the percentage of pectin used in the evaluation adversely affected all sensory parameters. While the cake with 15% pectin-coated cellulose received a total score of 80.4 out of 100 and might be acceptable particularly after formula optimization, the cake

15% PECTIN WITH TYPE D CELLULOSE



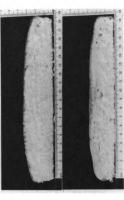


Figure 18. Pictures of cakes containing pectin coated cellulose

containing 30% pectin-coating on the cellulose received a total sensory score of 61.1. These cakes were characterized as being compact, gummy, soggy, dough-like and slightly grey.

Quality and Nutritional Significance of High Fiber Cakes

Forsythe et al. (1976; 1978) studied the effects of some of these cereal and cellulose products on laxation and cholesterol metabolism in the rat. The basal diet used was intended to approximate the "typical" American diet in that it contained 40% of the calories from fat, 40% from carbohydrate, and 20% from protein. This was supplemented with 1% cholesterol and 8% fiber analysed by neutral detergent fiber.

Cellulose Types A and B and CMC-coated cellulose reduced transit time by about one third and increased wet fecal mass by 2.5. Among the cellulose products only pectin-coated cellulose altered cholesterol levels. Both pectin coated products reduced cholesterol levels significantly although they did not significantly differ from each other (Forsythe, 1976).

Oat and wheat bran showed the greatest effect on laxation, with relative values of 5 compared to 3.2 for soybean hulls and 2.0 for corn bran. The commercial wheat bran was used for this study (Bennink, 1977). Particle size showed no effect on these laxation values. All of the cereal brans slightly elevated serum cholesterol

levels, although none of the values were significantly higher than the control (Forsythe, 1978).

Substituting 30% of the cake flour with cellulose or cellulose derivatives as was done in the present study would increase the fiber components of a 100 g piece of cake by 7.11 g. The addition of 30% commercial wheat bran, corn, oat, or soy bran would add approximately 2.84, 4.55, 1.34 and 4.05 g of fiber respectively.

All of the cakes in the present study made from wheat bran were acceptable at the 30% level of substitution and did not differ significantly from the control on total sensory points. There was also no significant difference found in total sensory points due to variety of wheat. Although objective measurements pinpointed significant differences in color due to the use of wheat bran and the type of wheat bran, the taste panel indicated that the colors of cakes containing all of the wheat brans were acceptable. It had been believed that the bran from white wheat would form a more acceptable product due to the lighter coloration. Thus, any of these wheat brans could be used as a source of dietary fiber.

Cakes made from corn bran had the largest volume of any cake. The main objection to this cake was that it tasted like corn. Therefore, these cakes showed lower flavor scores than the cakes made with wheat bran or the control. It must be noted, however, that the flavor

scores tended to increase as the panel became acclimated to the taste. Moreover, these cakes received an average of 81 total points compared to 91 for the control cakes.

The cakes made from soy and oat bran would probably be unacceptable due to low flavor scores. It is possible that these low scores could be due to oxidative rancidity since these brans had been stored for some time at room temperature. More studies are needed in this area to identify the factors leading to the low flavor scores observed.

All of the layer cakes with the various types cellulose or CMC-coated cellulose were of high quality and such products are feasible carriers of these dietary fibers which contribute to laxation. Cakes with pectin-coated cellulose had the greatest hypocholesterolemic effect (Forsythe et al., 1976). Formula modifications could be attempted to improve the quality of these cakes. In addition, lower levels of pectin-coating of the cellulose should be tested determine if they also produce a decrease in serum cholesterol and at the same time may be more acceptable in product formulation.

Chemical Composition

Fiber Data

The quantities of the \mathbf{v} arious fiber components in bran and flour samples are shown in Tables 21 and 22. The

S

Dietary fiber components of wheat brans 1

Table 21.

Source	Water Soluble Hemicellulose (%)	Pectin	Water Insoluble Hemicellulose (%)	Cellulose	Lignin ²
Hard Red					
Commanche	.46±.27	.75±.39	30.34±5.99	9.17±.96	4.35±.21
Shawnee	.53±.46	.714.39	25.41±2.41	8.58±.62	4.72±.68
Soft Red					
Oasis	.52±.21	.57±.28	24.90±3.45	7.79±.42	5.12±1.25
Arthur	.50±.26	.904.25	27.07±2.72	8.44±1.09	4.19±.14
Soft White					
Ionia	.45±.27	1.68±.65	24.20±2.49	5.98±2.22	3.90±.47
Yorkstar	.54±.32	.77±.48	24.29±2.48	6.82±1.16	3.65±.48

 $^{1}{\sf Means}$ and standard deviations are based on 4 replications

 2 3 replications

Dietary fiber components from various sources

Table 22.

Source	Water Soluble Hemicellulose (%)	Pectin (%)	Water Insoluble Hemicellulose (%)	Cellulose	Lignin (%)
Control Flour	41.±19	72.±44.	2.73±1.03	2.04≩.52	.53₹.25
Commercial Wheat Bran	88.±μμ.	.61±.54	28.49±4.38	8.40±1.19	5.35±1.78
Corn Bran	.30±.21	09.±95.	45.62±4.38	14.99±1.49	2.02±.07
Oat Hulls	.93±.56	1.11±.21	8.20±1.24	2.92 + .86	3.36±.11
Soy Bran	.89±.25	6.92±.89	17.57±3.31	36.19±4.69	3.10±.33

 1 Mean and standard deviations are based on $^{\mu}$ replications

²³ replications

 $³_2$ replications

control flour contained the lowest quantities of most fiber constituents. Physical separation methods have shown the water soluble pentosans content of wheat endosperm after enzymatic starch digestion to be between .4 and .6%. Fifty to sixty eight percent of this was carbohydrate in nature (Lineback et al., 1977). Patil et al. (1975a) found that the wheat endosperm contained .48% pentosans of which 16.4% was protein. D'Appolonia and MacArthur (1975) found .58 to .72% pentosans of which 20 to 30% was protein. Lin and Pomeranz (1968) found .38 to .58% pentosans with 17 to 23% protein. The current study showed .41% water soluble carbohydrates.

Physical separation methods have shown that the endosperm contains 2 to 3% water insoluble hemicelluloses (Neukom et al., 1962). Soughgate's fractionation scheme listed 2.8% water insoluble hemicelluloses (Southgate, 1977) while distillation of furfural components gave values of 1.8 and 2.4% hemicellulose (Fraser et al., 1956; Fraser and Holmes, 1957). These compare to 2.7% obtained in the current study. Cellulose and lignin values obtained in this study (2.2 and .5%) were higher than those listed by the previously mentioned studies (.6-.8%; .03%). Cellulose values obtained in this study are probably high since most of the hemicellular material remained in the cellulose extraction.

The water insoluble hemicellulose contents of the bran samples in the current study ranged from 24 to 30%. Distillation of furfural components and Southgate's fractionation scheme (Southgate, 1977) showed results of 26 and 35% respectively. Data by McConnel and Eastwood (1974) using Southgates method listed 25-28% water insoluble hemicelluloses. Data of Adam's cited by Hlynka (1964) listed 30% water insoluble hemicelluloses.

Cellulose contents of the wheat brans in the current study ranged from 6 to 9%. Rasper (1978) treated an acid detergent fiber residue with 72% sulfuric acid and obtained values of 8.6 to 12.4% cellulose. Southgate (1978) found 8.6% cellulose by his fractionation scheme. McConnel and Eastwood (1974) found 9-11% cellulose. Adams found higher values in the range of 25% cellulose (Hlynka, 1964). The lignin values of this study (3.6 to 5.4%) were again higher for the wheat bran samples than previously found (1.7 to 3.4%) by Rasper and Southgate. However, McConnel and Eastwood (1974) using Southgates method found 4.6 to 5.8% lignin. Adams values showed 5.6% lignin.

The corn bran of this study was found to contain 45.6% water insoluble hemicelluloses, 15% cellulolse and 2% lignin. This compares to 47-67% hemicellulose, 14-23% cellulose, and .2 to .7% lignin (Schaller, 1977; Rasper, 1978).

Oat hulls were found to contain 8% water insoluble

hemicellulose, 3% cellulose and 3% lignin. This compares to previous results of 35% cellulose and 6.7% lignin (Rasper, 1978). Obviously the samples were not milled alike. Soybean hulls contained 36% cellulose and 3% lignin. This compares with 41% cellulose and 3% lignin (Rasper, 1978).

Statistical analyses on the wheat brans showed significant differences between pectin, water insoluble hemicelluloses, cellulose, and lignin content (Table 23). differences seen in pectin were between red versus white brans and the two white wheat brans. differences were due to a much higher pectin content found for Ionia. Cellulose content showed significant differences between the hard and soft wheat brans and between the red versus white wheat brans. The hard wheat samples were higher than the soft and red higher than the white wheat brans in cellulose content. Insoluble hemicelluloses showed significant differences between the two hard red wheat brans and two soft red wheats as well as between the hard versus soft and red versus white wheat brans. Lignin content differed between the red and white wheat brans, and the commercial source compared to the rest of the wheat brans. The red brans were higher than the white brans in lignin contents.

More significant differences in fiber component proportions appeared in the comparison of the control

Analyses of variance for major fiber constituents

Table 23.

			F Value		
Source	Cellulose	Water Soluble Hemicellulose	Water Insoluble Hemicellulose	Pectin	Lignin
Block	5.27*	203.48**	38.46**	6.88	6.52*
Variable	145.78**	295.53***	227.67***	81.20***	126.83***
Control vs rest	116.14**	16.33***	845.24**	21.64**	68.13**
Wheat vs non-wheat	336.89**	83.31**	25.03**	195.89***	36.35**
Corn vs Oat vs Soy	481.04**#	***90° h6	674.03***	287.27###	3.17
Commercial vs other wheats	ης.	2.05	9.18**	1.67	5.41*
Hard versus soft	5.93*	.11	18.06**	1.89	.67
Red vs white	#96·ħ	.11	5.38*	5.62*	2.66*
Commanche vs Shawnee	.30	.62	21.60***	.01	44.
Oasis vs Arthur	.35	00.	4°19*	1.24	3.65
Ionia vs Yorkstar	.59	4.87*	00.	9.51**	.21

Significant at the 5% level of probability

* Significant at the 1% level of probability

** Significant at the 0.1% level of probability

versus all the other types; the wheat versus the non-wheat brans. The control versus the rest and wheat brans versus non-wheat brans differed in pectin, insoluble hemicellulose, soluble hemicellulose, cellulose, and lignin content. It has been hypothesized that wheat bran may contain more water soluble pentosans than the endosperm (Preece and MacKenzie, 1972). This was found to be true. The three non-wheat brans differed in all fiber component content. Corn bran contained the greatest amount of water insoluble hemicellulose, while soy bran contained the greatest quantity of cellulose. Oat bran was low in both of these.

The hemicelluloses were also measured as hexoses. pentoses, and uronic acids (Tables 24 and 25). The minor constituents of the hemicelluloses in wheat bran showed no significant differences (Table 26). Analysis of variance of the other cereals showed significant differences both minor constituents. These differences occurred in comparisons of the control versus any bran and among the non-wheat brans. The percentages of hexoses and uronic acids to pentose concentration are shown in Tables 27 and 28. The water insoluble hemicelluloses were divided into the two extractions. The first extraction showed values of 10-13% uronic acids and 21-25% hexoses for the wheat brans. Corn and oat hulls were slightly lower in uronic acids and slightly higher in hexose content. Soy brans

Composition of the hemicellular material 1 in wheat brans

Table 24.

	Water Soluble	e Hemicellulose	Water I	Insoluble Hemi	Hemicellulose
Source	Pentoses (%)	Uronic Acids (%)	Pentoses (%)	Hexoses (%)	Uronic Acids (%)
Hard Red					
Commanche	.22±.14	.26±.15	23.87±4.28	4.17±.90	2.30±.60
Shawnee	.30±.25	.23±.21	20.18±2.23	3.49±.48	1.74±.08
Soft Red					
Oasis	.31±.12	.19±.09	19.90±3.87	3.21±.52	1.79±.23
Arthur	.25±.10	.25±.23	21.45±1.80	3.56±.34	2.06±.35
Soft White					
Ionia	.35±.17	.08±.13	79.73±2.50	2.85±.20	1.62±.21
Yorkstar	.35±.20	.20±.13	19.53±2.48	3.03±.81	1.68±.18

 1 Means and standard error of the means are based on 4 replications

Composition of the hemicellular material of various plants

Table 25.

	Water Solubl	uble Hemicellulose	Water I	Water Insoluble Hemicellulose	.cellulose
Source	Pentoses (%)	Uronic Acids (%)	Pentoses (%)	Hexoses (%)	Uronic Acids (%)
Control Flour	.20±.11	.20±.10	2.19±.34	.48±.25	.06±.13
Commercial Wheat Bran	.22#.22	.23±.17	22.72±4.20	3.55±.38	2.22±.77
Corn Bran	.18±.09	.12±.12	34.38±4.24	7.61±2.63	3.70±1.37
Oat Hulls	.40±.20	.53±.38	7.12±1.37	.71±.11	.37±.11
Soy Bran	.37±.10	.52±.16	14.61±3.64	1.51±.24	1.45±.26

 $^{1}\mathsf{Means}$ and standard error of the means are based on 4 replications

26. Analyses of variance of the individual components of the hemicelluloses Table

			F Values		
	Water Soluble	le Hemicellulose	Water	Insoluble	Hemicellulose
Source	Pentose	Uronic Acids	Pentose	Нехозе	Uronic Acids
Block	11.85**	*60.7	2.44	.71	1.50
Variables	1.67	4.08	**19.6	3.77	3.03
Control vs rest	2.29	.67	17.73***	6.62*	7.38*
Wheat vs non-wheat	.56	15.67***	.26	.02	00.
Corn vs Oats vs Soy	4.30*	10.22**	38.19**	*** 77. 71	** 10.89**
Commercial vs wheats	1.63	60.	1.00	.50	.51
Hard vs soft	1.17	1.06	60.	.08	. 15
Red vs white	1.24	1.13	.08	. 14	. 15
Commanche vs Shawnee	.80	. 10	.08	.01	.02
Oasis vs Arthur	. 43	.31	.10	. 18	. 23
Ionia vs Yorkstar	00.	1.33	.95	69.	60.
1					

¹ Taken from the first hemicellulose extraction.

* Significant at the 5% level of probability.

** Significant at the 1% level of probability.

*** Significant at the 0.1% level of probability.

15.85 16.02 15.57 Uronic Acids Water Insoluble Hemicellulose 12.18 13.02 12.41 11.93 11.22 10.01 E Ratio of uronic acid and hexose to pentose content 26.07 31.41 25.90 23.72 25.25 20.10 H2 Hexoses **%** 23.62 20.89 24.30 23.84 22.77 25.19 E Water Soluble Hemicellulose Uronic Acids (%) 118.36 77.88 98.08 58.32 61.77 Commanche Soft White Yorkstar Table 27. Shawnee Source Hard Red Soft Red Arthur Oasis Ionia

Ratio of uronic acids and hexose to pentose content

Table 28.

	Water Soluble Hemicellulose	M	Water Insoluble Hemicellulose	e Hemicellul	o s e
Source	Uronic Acids	He He	Hexoses (%)	Uronic	Uronic Acids
		H 1	H2	H 1	HZ
Control Flour	101.19	48.77	1259.	0.00	233.
Commercial Wheat Bran	105.15	24.16	45.45	12.24	20.25
Corn Bran	76.51	24.54	32.21	11.35	16.93
Oat Hulls	137.77	38.81	27.61	11.57	21.48
Soy Bran	148.06	38.07	44.85	30.05	49.15

contained a greater proportion of both uronic acids and hexoses. Legumes have previously been found to contain more uronic acids than cereals (Pigman and Horton, 1970). Most of the brans tended to have a slightly larger proportion of hexoses and uronic acids in the second extraction. Values of uronic acids found for corn bran (11 and 17%) are similar to those found by Seckinger et (1960). The minor constituents of wheat brans are al. also similar to those found by Southgate (1977). He found uronic acids and 27% hexose. The water insoluble hemicelluloses of wheat flour have been found to contain approximately 41% hexoses (Cole, 1967) when fractionated on DEAE-cellulose columns. Southgate listed 733% hexose and 83% uronic acids. The results of this study also showed larger amounts of hexose and uronic acids.

The uronic acid content of water soluble hemicelluloses has not been studied. The values found by this study were very high, showing values as high as the pentose content. It is probable that some of this was water soluble pectic materials.

Non-fiber Components. The non-fiber components of the brans and flour samples are shown in Table 29. Starch contents ranged from 2% for soy bran to 66% for the control flour. Fat contents for the control and corn bran were lower than the rest which showed similar values. Moisture and ash contents were similar between samples

Table 29. Non-fiber component levels in the bran and flour samples

Starch %	Fat %	Moisture %	Ash %	Protein %
65.70	1.24	8.56	5.10	8.32
16.05	3.99	8.33	7.31	13.92
16.50	4.38	7.69	7.47	13.07
15.31	8.94	8.54	7.14	15.35
11.41	5.07	8.67	8.44	15.29
10.47	5.16	10.02	7.71	13.46
15.55	4.45	9.34	7.62	13.76
13.89	5.52	7.94	7.25	14.50
19.32	1.46	6.57	1.08	5.82
1.95	3.95	6.25	5.20	13.17
21.04	4.76	7.49	5.59	27.92
	\$ 65.70 16.05 16.50 15.31 11.41 10.47 15.55 13.89 19.32 1.95	\$ \$ 65.70 1.24 16.05 3.99 16.50 4.38 15.31 8.94 11.41 5.07 10.47 5.16 15.55 4.45 13.89 5.52 19.32 1.46 1.95 3.95	% % 65.70 1.24 8.56 16.05 3.99 8.33 16.50 4.38 7.69 15.31 8.94 8.54 11.41 5.07 8.67 10.47 5.16 10.02 15.55 4.45 9.34 13.89 5.52 7.94 19.32 1.46 6.57 1.95 3.95 6.25	% % % 65.70 1.24 8.56 5.10 16.05 3.99 8.33 7.31 16.50 4.38 7.69 7.47 15.31 8.94 8.54 7.14 11.41 5.07 8.67 8.44 10.47 5.16 10.02 7.71 15.55 4.45 9.34 7.62 13.89 5.52 7.94 7.25 19.32 1.46 6.57 1.08 1.95 3.95 6.25 5.20

except that corn bran had a much lower ash content.

Protein content was lowest in corn bran and highest in the oat bran.

Recoveries. Recovery data were obtained by adding the quantities of hexoses, pentoses, and uronic acids measured in each extraction to the fat, protein, moisture, ash, and lignin values. Final recovery values are shown in Table 30. Values ranged from 95.9% to 104.7%. Both of the soft white wheats had the lowest values while the recovery data for soy bran showed recovery values much higher than any other bran. Its high value was due to the fact that it has a substantial amount of total uronic acids. acids interfere with the pentose reaction causing artificially high values. Interference due to uronic acids is approximately 1/2 its value. Therefore had this been subtracted out recovery values for soy would been 4% lower. The wheats would have shown recovery values 1.5% lower, corn would have been reduced by 2% and oats by .6%. These values were not subtracted off because it was not felt that the uronic values were accurate enough.

Comparison of Total Fiber Values. Total fiber values obtained by this method were compared to data obtained on the same samples by the enzymatic neutral detergent (ENDF) fiber method (Table 31). Values were in general very

Table 30. Average percent recovery obtained for bran and flour samples

Source	Recovery %
Hard Red Wheat Bran	
Commanche	101.7
Shawnee	100.3
Soft Red Wheat Pran	
Oasis	102.1
Arthur	98.6
Soft White Wheat Bran	
Ionia	95.9
Yorkstar	97.0
Commercial Wheat Bran	103.3
Corn Bran	102.5
Soy Bran	104.7
Oat Hulls	101.8
Control	97.9

Table 31. Comparison of total fiber by two methods

		Method
Source	NDF/ENZ %	Fractionation %
Hard Red Wheat Bran		
Commanche	44.3	45.1
Shawnee	36.5	40.0
Soft Red Wheat Bran		
Oasis	38.0	38.9
Arthur	39.7	41.1
Soft White Wheat Bran		
Ionia	37.5	36.2
Yorkstar	37.0	36.1
Commercial Wheat Bran	39.6	43.3
Corn Bran	62.3	63.5
Soy Hulls	56.2	64.7
Oat Bran	19.0	16.5

close indicating that this method is fairly accurate in its estimation of fiber content. Most of the values obtained by the fractionation method are slightly higher than those obtained by ENDF, however, slightly lower values were obtained for the soft white wheats, which had shown low recovery values, and oat bran. The values obtained for soy bran by the fractionation procedure were 8.5% higher than values obtained by ENDF. This is due in part to the high recovery values and in part by the fact that ENDF does not include pectic substances or water soluble hemicelluloses. This is also the reason for the slightly higher values obtained for the other brans. Other research using ENDF methods have shown 38-45% fiber for wheat brans, 93.7% for corn bran, 8.6% for oat hulls, and 67% for soy hulls (Rasper, 1978).

Relationship between Fiber Components and Cake Quality

Simple Correlations. Several significant simple correlations were evident between fiber components and cake parameters. Table 32 shows those correlations which were significant at P<.01. Those cake parameters involved were tenderness, volume, cell size, cell wall thickness and grain. Viscosity correlated at P<.1 while sensory tenderness, moistness, softness, objective moisture and uniformity, although showing significant differences among cakes, showed no significant simple correlations.

Simple correlations 1 of fiber components with cake parameters

Table 32.

		Cake	Parameters	e r s	
Component	Tenderness	Volume	Cell Size	Cell Thickness	Grain
	C L	i	-		
water Soluble Hemicellulose (WSHEM) Water Insoluble Hemicellulose (HEM)	04. - 74.	ן טרי - אר	1.04	ا / . ا رک	0 4
ectin (PECT)	•))	44	96	
ignin (L1	.58				
SHE	56	45	99	73	74
田田	.37	19.		. 43	
			42	36	
IG Sq	.59			4.	
ellulo			37		
EL X HEM		. 42			. 41
EL x PEC			0 7 -	34	
EL x W			39	,	
EM x L	.79		.37	.63	
EM X WSHE	.55			•	
工		64	39		
ECT x LI			43	34	
ECT x WS			L + -	- ¥3	

1Significant at p <= .01

Previous research has shown that water insoluble hemicelluloses improve cake volume and internal characteristics (Baldi et at., 1965; Donelson and Wilson, 1960a). This was found to be the case here, where insoluble hemicelluloses were positively correlated with increased volume, increased tenderness, and thinner cell walls. Water soluble hemicelluloses have been found to decrease cake volume (Donelson and Wilson, 1060a). Again this was found to be true. Soluble hemicelluloses were correlated with decreased tenderness, decreased volume, larger cells and thicker cell walls.

Pectin correlated with larger cells and thicker cell walls. The lower levels of pectin found in the brans did not show decreased volume or increased tenderness as did the high levels of the pectin coated variables of the cellulose study. However, since only one variable (soy) contained a substantial amount of pectin, and the other pectin values were not considered accurate, these correlations should not be considered conclusive.

Cellulose by itself showed no significant correlations while cellulose correlated only with larger cells. The study incorporating cellulose into cakes also showed few significant effects. A difference between the mechanically ground and hydrolysed cellulose was seen. The mechanically ground cellulose showed a higher volume and viscosity. This may be due to the hemicellular

content which has been found to be up to 15% (Lang and Briggs, 1976). Brys and Zabik (1976) showed few significant differences until substitution levels greater than 40%. The correlations which were significant for the component squared tended to be the same as those exhibited by the component itself.

Several interactions were seen between fiber components. In most cases the interactions were between components which showed the same correlations. Several interactions also appeared where one component had shown a correlation and the other had not. These were seen between CELXHEM, WSHEMXLIG, and PECTXLIG. In these cases the correlation followed that seen by the hemicellulose or pectin component. In only one case was there interaction between components which had shown the opposite simple correlations. This was HEMXWSHEM for In this case the correlation followed that of tenderness. the water insoluble hemicellulose.

These simple correlations cannot be thought of as indicating the absolute effect of these substances on the parameter measured because of possible interactions between component levels. For instance, lignin showed a correlation with increased tenderness. This effect seems unlikely because lignin is often associated with vegetable toughness. However, lignin content may be correlated with the content of some other component. In fact there is a

significant correlation (P<.01) with the water insoluble hemicelluloses which show the same effect. Lignin content has been found to follow hemicellulose and cellulose contents (Gaillard, 1962).

Batter viscosity has been implicated as an indicator of final cake quality. Studies have shown that more viscous batters result in cakes which have finer texture with more evenly distributed air cells (Collins and Sunderline, 1940)

In this study viscosity showed no correlation with volume, tenderness, or cell size. Significant correlations did exist, however, between moistness and cell uniformity. While objective moisture showed a negative correlation, sensory moisture and cell uniformity showed positive correlations.

Tenderness was positively correlated with size and cell wall thickness. Objective moisture was negatively correlated with cell uniformity, sensory moisture and softness. Uniformity showed a positive correlation with sensory moistness and softness, while uniformity was positively correlated with cell size.

<u>Prediction Equations</u>. Prediction equations were calculated using fiber components as the independent variables. The following parameters could be predicted by fiber content: volume, tenderness, viscosity, grain, cell size, and thickness of cell walls (Table 33). Viscosity

Table 33. Prediction equations for cake parameters

Parameter		Equation	R so
Tenderness	Y =	-1.89900 WSHEM + 1.7685 WSHEMSQ 0016 HEMPECT0020 HEM LIG + 1.9649	.77
Volume	Y =	.0004 HEM SQ1390 LIG WSHEM + 10.1433	.49
Viscosity 1	Y =	-59.42 CEL - 200.57 LIG + 48.20 HEM + 4853.86 WSHEM - 1773.43	. 17
Cell Size	Y =	-3.1268 WSHEM + 10.0681	. 4 1
		.0325 CEL + .3535 LIG0234 HEM - 5.2528 WSHEM + 10.7395	.67
Grain	Y =	-6.2064 WSHEM0023 HEMSQ + .1225 HEM WSHEM + 16.9894	.68

¹This equation does not include the oat data

was significant at P<.1, while the others were significant at P<.0005.

These equations start with one component and perform partial correlations of the other components with the component chosen.

How well the equation predicts the parameter measured is shown by the R^2 value. This tells how much of the variation in quality can be predicted by the equation. Therefore, 77% of the variation in tenderness can be predicted by this equation. Between 40 and 60% of the variation in other cake parameters was predicted by the fiber components. These prediction values are good since they indicate that over 50% of the variation in cake quality can be predicted by fiber components alone. Viscosity was the exception. Only 17% of the variation could be explained. Pectin and hemicelluloses are known to have a high water binding capacity (Eastwood, 1973; Jelaca and Hlynka, 1971) and hemicelluloses have been shown to increase viscosity (Baldi et al., 1965). Also, high levels of cellulose (>20% of the flour) increase viscosity (Brys and Zabik, 1967). The high levels of cellulose and pectin in the cellulose study both caused an increase in viscosity. However, although most of these parameters figured into the prediction equation. prediction was still quite poor. It must be noted that all of the brans increased viscosity, even oats which are

low in most fiber components. Therefore something other than fiber is causing the large effect seen. Protein and starch values were also put into an equation along with total fiber, but did not help the predictability. Particle size has a large effect on viscosity and may have contributed to the variations seen.

Water soluble or insoluble hemicelluloses always figured into the prediction equations, indicating that these are important indicators of cake quality. Water soluble hemicellulose components were of major importance to tenderness and cell size while water insoluble hemicellulose was the most important factor to volume. Taking this factor out would have reduced predictibility from .49 to .24. Both hemicellulose fractions were important to grain, appearing both singly and in interactions. Pectin, cellulose, and lignin most often occurred as interactions with one of the hemicelluloses indicating that these are of lesser importance.

Energy Consumption

There has been concern expressed that bran usage may not be feasible due to the extra energy which may be necessary to grind the bran. The number of grindings necessary to reduce the bran was recorded to determine if one type of bran would be more resistant to grinding than another. No difference was seen using the cyclone mill between hard or soft wheat brans.

SUMMARY AND CONCLUSIONS

Layer cakes were prepared with 30% of the flour with various cereal brans and cellulose substituted products to evaluate the effect of different sources fiber on cake quality. Statistical analyses of the objective measurements of the control cakes versus the other cakes containing bran showed significant differences in most parameters including tenderness, viscosity, all color measurements. Volume indices showed significant differences in this comparison. Sensory showed very few significant differences when evaluation the control was compared to the cakes containing wheat bran, however the cakes containing nonwheat bran scored lower than the control on most sensory evaluations.

Objective measurements of the cakes containing wheat versus non-wheat bran showed significant differences in all measurements but volume. Batters made from non-wheat bran were more viscous, and the cakes less tender and more moist. Sensory scores indicated that the cakes made from non-wheat bran were significantly less acceptable.

Although as a group the cakes containing non-wheat bran behaved significantly different from the other cakes, wide variations were seen among these cakes for most

objective and sensory scores. The individual wheats in each varietal category showed few differences.

All of the cakes made from wheat bran were acceptable at the 30% level of substitution. Cakes made from corn bran had the largest volume but was disliked due to its corn flavor. Cakes made from soy and oat bran would probably be unacceptable due to low flavor scores.

Analyses of subjective and objective data indicated few significant differences among the cakes containing any cellulose type or cellulose coated with CMC. Parameters which were significantly affected included increased batter viscosity with the addition of cellulose and alteration of some of the color measurements.

When mechanically ground cellulose was compared to chemically hydrolysed cellulose differences were seen in viscosity, volume, and symmetry indices as well as several sensory parameters. Mechanically ground cellulose formed the better product.

Substituting pectin-coated cellulose products caused increased batter viscosity, decreased cake volume, and overall lower sensory evaluations. All of the cakes except those containing pectin were acceptable.

The technique chosen for separate fiber component analyses worked fairly well in most cases, and provided information on the minor constituents present in the hemicellulose fractions. Total fiber measured by this

method agreed well with values obtained by Neutral Detergent Fiber. Problems with the method used included partial enzymatic digestion of the hemicellular material and difficulties with pectin analysis due to impure enzyme and large standard deviations in the uronic acid test.

These methods quantitated the amount of water soluble hemicellulose, water insoluble hemicellulose, cellulose, pectin, and lignin. The wheat brans which had caused only minor differences in cake quality were also similar in fiber content, showing approximate values of .5% water soluble hemicellulose, .8% pectin, 26% water insoluble hemicellulose, 8% cellulose and 4% lignin. Significant differences were seen among the wheat brans in pectin, water insoluble hemicellulose, cellulose, and lignin content.

The control varied from the other brans and the wheat brans from the non-wheat brans in pectin, insoluble hemicellulose, cellulose, and lignin content. The nonwheat brans differed in the content of all of the fiber constituents. Oat hulls were the lowest in most fiber components. Soy bran contained the greatest amount of pectin and cellulose, while corn bran contained the largest amount of water insoluble hemicellulose.

The minor constituents of the hemicelluloses in wheat bran showed no significant differences, while the other cereal brans showed significant differences in both minor

constituents when compared to the control, wheat bran or among themselves.

The ratio of uronic acid compared to pentose was much higher in the water soluble hemicelluloses compared to the water insoluble hemicelluloses.

Simple correlation data and prediction equations for cake parameters using fiber components as the independent variables were calculated. The results showed that fiber components correlate and can be used to predict many of the variations in cake quality noted. The hemicelluloses appeared to have a large effect on cake quality, while pectin and cellulose at the levels which occured in the cereal brans had little effect on most parameters.

SUGGESTIONS FOR FURTHER RESEARCH

- 1) Different enzyme systems should be used in an effort to minimize extraction of hemicellular material.
- 2)A more pure enzyme or enzyme purification should be tried to eliminate problems in analysing pectin content and the uronic acid test should be altered to obtain more consistent results.
- 3) The hexose fractions of the separate extractions should be fractionated into individual sugars to further analyse the minor constituents in the fractions and pinpoint possible contamination of one fraction with remnants of another.
- 4) Bran samples from more seeds should be analysed chemically and in the cake system to further pinpoint component level effects.
- 5) This work could be carried out in other systems, particularly the bread system.



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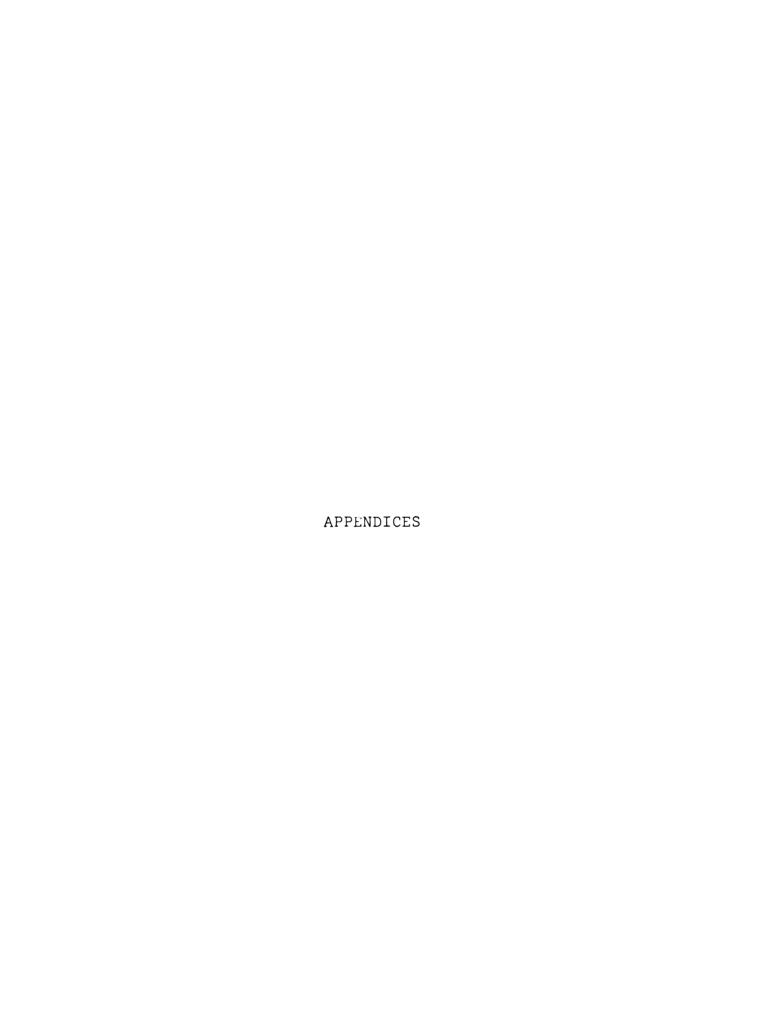
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 Note on layer cakes containing 30 to 70% wheat bran.

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Appendix I

LAYER CAKE SCORE CARD

Α.		s (30 points) Uniformity (10 points) (a) Even (normal) (b) Slightly uneven (c) Uneven	10 6 2
	2.	Size (10 points) (a) Dense (normal) (b) Close (c) Slightly open or slightly compact (d) Open or compact	10 8 6 4
	3.	Thickness of Walls (10 points) (a) Thin (normal) (b) Slightly thick (c) Thick	10 6 2
В.	1. 2.	n (16 points) Silky (normal) Harsh Coarse (cornbread)	16 10 8
С.		ture (34 points) Moistness (10 points) (a) Gummy (b) Moist (normal) (c) Slightly dry (d) Dry	6 10 8 4
	2.	Tenderness (14 points) (a) Very tender (normal) (b) Tender (c) Sightly tough (d) Tough	14 12 10 4
	3.	Softness (10 points) (a) Soft (normal) (b) Slightly firm (b) Slightly firm (c) Firm	10 8 8 4
D.	1. 2.	nb Color (10 points) Acceptable Slightly unacceptable Unacceptable	10 5 0
Ε.	1. 2.	or (10 points) Acceptable Slightly off flavor Unacceptable (off flavors present)	10 5 0

Appendix II

Average results for the total water soluble extraction

Sample	Hexose (%)	Pentose (%)
Arthur	5.63	.59
Ionia	5.59	.88
Control	3.25	. 43
Oats	13.58	.56
Shawnee	6.26	.87
Oasis	6.05	.66
Soy	4.00	.82
Corn	2.18	.51
Yorkstar	7.46	.77
Commanche	5.47	.90
Commercial	6.52	.70

160
Average results for the water soluble hemicellular material

Sample	Hexose (%)	Pentose (1)	Uronic (%)
Arthur	. 40	.25	.25
Ionia	. 14	.35	.08
Control	. 24	.20	.20
Oats	. 40	.40	.55
Shawnee	.35	.30	.23
Oasis	.37	.31	.19
Soy	.33	.37	.55
Corn	.06	.18	.13
Yorkstar	.39	.35	.20
Commanche	.22	.22	.26
Commercial	.33	.22	.23

161
Average results for the starch extraction

Sample	Hexose (%)	Pentose (%)
Arthur	11.41	. 32
Ionia	16.05	.30
Control	65.68	.77
Oats	21.04	.35
Shawnee	15.55	.38
Oasis	15.31	.38
Soy	1.95	1.06
Corn	19.32	.37
Yorkstar	16.50	. 43
Commanche	10.47	.33
Commercial	13.89	.26

162
Average results for the protein extraction

Sample	Hexose (%)	Pentose (%)
Arthur	.58	.88
Ionia	.93	1.41
Control	1.93	.36
Oats	1.22	.84
Shawnee	.58	.95
Oasis	.65	1.09
Soy	1.63	1.29
Corn	. 64	.99
Yorkstar	.79	1.34
Commanche	.74	1.46
Commercial	.75	.96

163
Average results for the pectin extraction

Sample	Hexose (%)	Pentose (%)	Uronic (%)
Arthur	2.01	2.78	.90
Ionia	2.94	3.74	1.68
Control	2.47	0.00	. 44
Oats	3.23	3.32	1.11
Shawnee	2.18	3.08	.71
Oasis	1.85	1.43	.57
Soy	3.16	3.39	6.92
Corn	1.59	2.60	.56
Yorkstar	2.46	2.90	.77
Commanche	2.84	2.55	.75
Commercial	2.76	3.89	.61

164

Average results for the hemicellulose 1 extraction

Sample	Hexose (%)	Pentose (%)	Uronic (%)
Arthur	1.81	7.59	.91
Ionia	1.49	6.55	.85
Control	.19	.39	.00
Oats	.38	.97	.11
Shawnee	1.84	7.56	. 94
Oasis	1.43	6.85	.69
Soy	.69	1.80	.54
Corn	4.67	19.04	2.16
Yorkstar	2.22	8.83	.99
Commanche	1.94	8.20	1.00
Commercial	2.27	9.38	1.15

165
Average results for the hemicellulose 2 extraction

Sample	Hexose (%)	Pentose (%)	Uronic (%)
Arthur	1.75	7.39	1.15
Ionia	1.36	5.40	.77
Control	.29	.01	.05
Oats	.33	1.19	.26
Shawnee	1.66	5.28	.81
Oasis	1.78	6.87	1.10
Soy	.82	1.84	.90
Corn	2.94	9.11	1.54
Yorkstar	.87	4.30	.68
Commanche	2.24	8.58	1.30
Commercial	1.28	5.28	1.07

Average results for the cellulose extraction

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Sample	Hexose (%)	Pentose (%)	
Arthur	8.44	2.57	
Ionia	5.98	2.38	
Control	2.25	.72	
Oats	2.92	.52	
Shawnee	8.58	2.93	
Oasis	7.79	2.33	
Soy	36.19	5.32	
Corn	14.99	2.20	
Yorkstar	6.82	2.40	
Commanche	9.17	2.75	
Commercial	8.40	2.95	