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DEVELOPMENT OF LOW-DENSITY MATERIAL THROUGH CARBONIZATION OF PUFFED WHEAT

by

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SUMMARY OF RESULTS

Puffed wheat was successfully carbonized at temperatures between 232° and 260°C (450° and 500°F). A 1/2-in. bed of material required between 2 1/2 hr at the lower end of the temperature range and 20 minutes at the upper end. By reducing the air in contact with the material to a minimum, the time required for carbonization was reduced to 1 3/4 hr at the lower temperatures but not appreciably at the upper temperatures. Densities of 2.0 to 2.2 lb/cu ft were achieved. Carbonization was accompanied by shrinkages of 30 to 40 per cent.

INTRODUCTION

The Mineral Processing Division was approached by TDR Engineering Developments Limited, Toronto, Ontario, to do a study to establish parameters for carbonizing puffed wheat to "produce a new system of materials". Some work done by this company had shown that puffed wheat could be converted to carbon by heating. The product had retained the shape and approximate size of the original kernels but was, in essence, particles of cellular carbon. The company reported that the theoretical density of the largest carbonized particles would be less than 1 lb/cu ft and that a typical thermal conductivity (K) would be 0.2 Btu in./hr ft^2°F.

The company submitted seven bags, approximately 14 cu ft, of puffed wheat. Examination of the sample revealed a minor quantity of fine material ranging from fragments of puffed wheat down to dust, and that the sample was composed of two types of puffed wheat. The majority was of well-expanded kernels, primarily cream coloured. A part of the sample was of poorly to moderately expanded kernels, yellowish in colour. In some cases, the latter had agglomerated into small clusters. Only the plus 1/4-inch well-puffed wheat was investigated in this study. The puffed wheat kernels incorporated about 10 per cent of the original bran.

The goal of this study was to establish firing conditions under which the puffed wheat kernels would be rendered black, (complete carbonization) with minimum reduction in volume.

COMPOSITION

About 83 per cent of a wheat kernel is the endosperm. It is made up of cells filled with starch granules, with gluten between the granules and cellulose walls dividing the cells. The bran, which normally surrounds the endosperm, makes up 15 per cent of the kernel, and the wheat germ about 2 per cent. The chemical composition of wheat is within

the following ranges:

carbohydrates (starch and cellulose)	65 to 78 per cent
protein (gluten)	12 to 14 " "
water	10 to 12 " "
fat	1 to 8 " "
minerals	1 to 2 " "

Carbohydrates are composed of carbon, hydrogen, and oxygen. Gluten is composed of carbon, hydrogen, oxygen, nitrogen, and sulphur. The approximate relationships of the components by weight are 50 per cent carbon, 45 per cent oxygen, 5 per cent hydrogen, and minor quantitites of nitrogen and sulphur.

THERMAL GRAVIMETRIC ANALYSIS

A test was run in a Stanton thermo-balance to determine, through what temperature range and at what rate that gases are evolved from the puffed wheat. Several kernels were ground to minus 16 mesh and a representative 1.0000-g sample was placed in the thermo-balance. The sample was heated to 900°C (1650°F) at a rate of 6 Celsius degrees per minute. During this time, it was being continuously weighed, the weight being automatically recorded. The curve thus obtained on the recorder chart was replotted to indicate percentage weight loss. The weight-loss curve is shown in Figure 1.

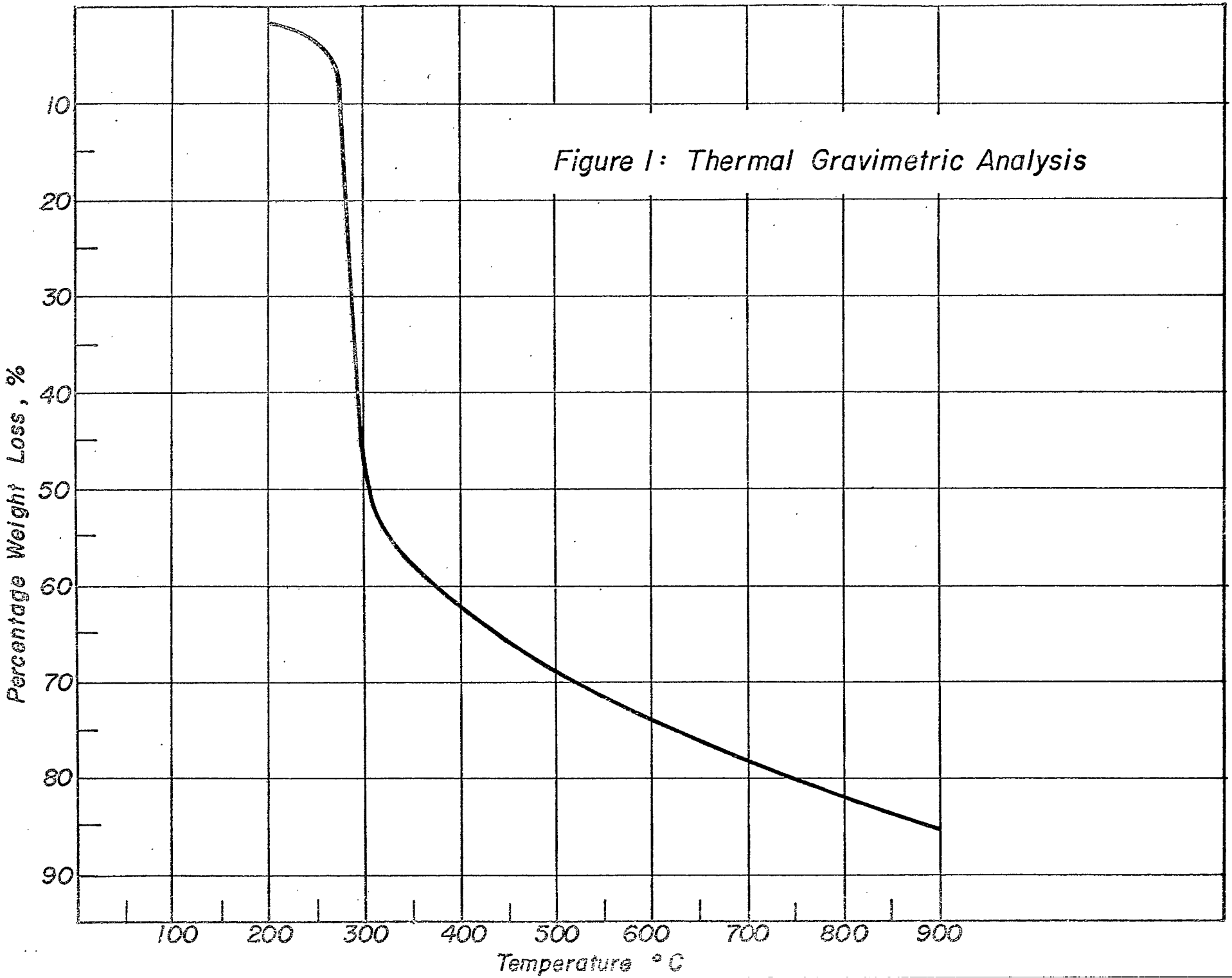
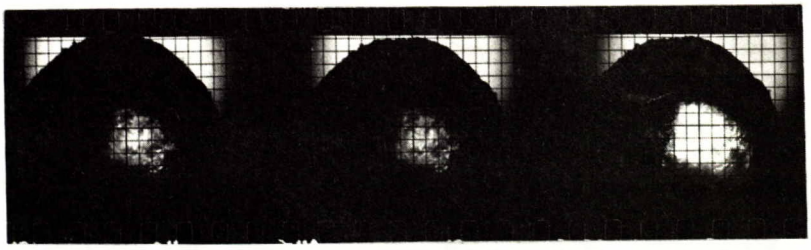


Figure I: Thermal Gravimetric Analysis
Percentage Weight Loss, %
10
20
30
40
50
60
70
80
90
100
200
300
400
500
600
700
800
900
Temperature °C

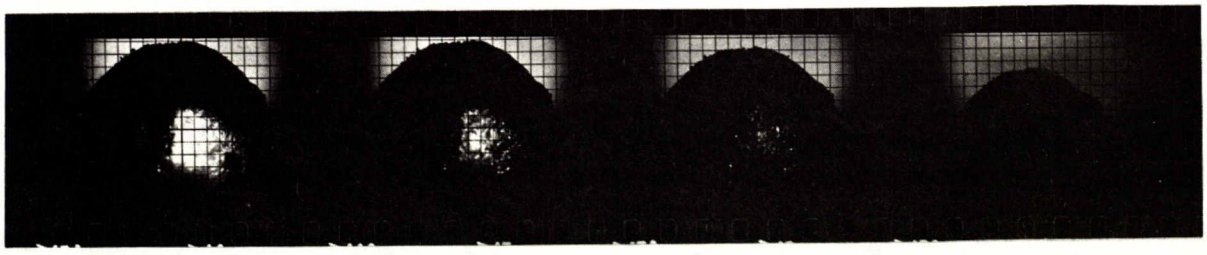
HEATING MICROSCOPE

Kernels and/or fragments of kernels were heated in a Leitz heating microscope to see the effect of heat on puffed wheat. The electrically heated furnace of the microscope is 3/4 in. in diameter by 4 in. long, the zone of maximum temperature being 1 in. long. The specimen was inserted into the hot zone of the furnace on a specimen holder attached to the end of the thermocouple that measured the furnace temperature. The ends of the furnace were closed to prevent passage of air or turbulence within the furnace. During the heating, the specimens were photographed against a grid.

Heating was done in two atmospheres; (1) the air which is normally contained in the furnace, and (2) a nitrogen atmosphere. The results were compared to determine if the carbon in the specimen would be oxidized in the air atmosphere. Different rates of heating were also employed. The condition of the specimen was examined through the eye piece at frequent intervals as it was heated and one of the photographs was taken as soon as any change in volume was observed. The photographs are shown in Figures 2 to 5.

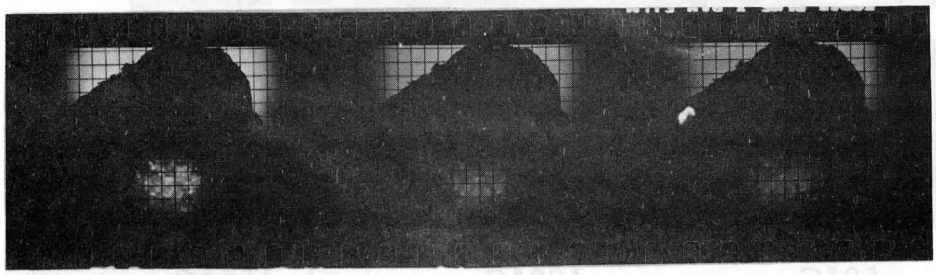


50°C 120°F | 100°C 210°F | 150°C 300°F



210°C 410°F | 240°C 465°F | 265°C 510°F | 275°C 525°F

Figure 2: Air Atmosphere
20-minute heating period.



20°C 70°F | 100°C 210°F | 145°C 295°F

200°C 390°F | 225°C 435°F | 250°C 480°F | 275°C 525°F

Figure 3: Air Atmosphere
55-minute heating period.

20°C
70°F

70°C
160°F

100°C
210°F

150°C
300°F

200°C
390°F

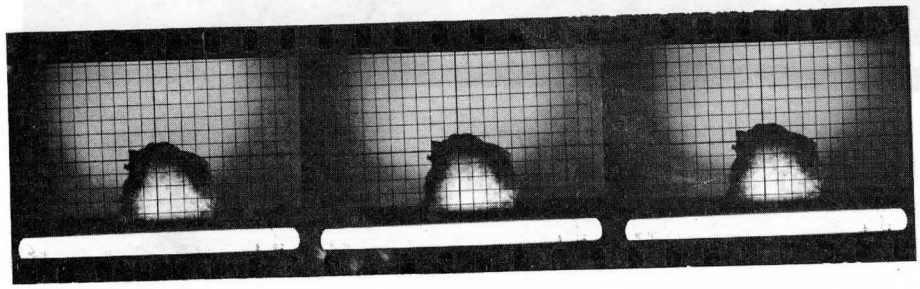
230°C
445°F

250°C
480°F

265°C
510°F

280°C
535°F

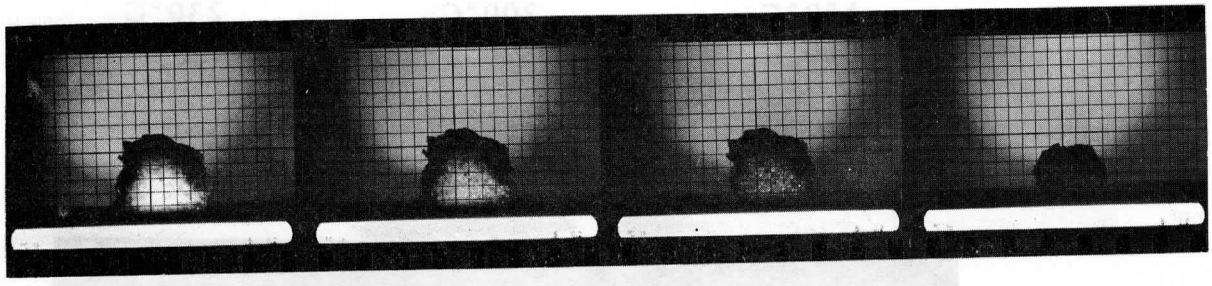
Figure 4: Nitrogen Atmosphere
25-minute heating period.



25°C
75°F

100°C
210°F

200°C
390°F



230°C
445°F

250°C
480°F

280°C
535°F

290°C
555°F

Figure 5: Nitrogen Atmosphere
40-minute heating period.

TUBE FURNACE TESTS

These tests were performed in a Lindberg electrically heated tube furnace that incorporated a porcelain tube (internal diameter, 1 inch) Charges of 8 to 10 kernels of puffed wheat were inserted into the furnace in a porcelain combustion boat 3 x 1/2 x 1/2 in. The thermal gravimetric analysis showed that weight loss is greatest between 250° and 300°C and the heating-microscope study showed that shrinkage began between 225° and 280°C. The temperatures used in the tube furnace were between 200° and 260°C (390° and 500°F).

For each test firing, the charge was pushed rapidly into the hottest zone of the tube and removed after a predetermined time interval. Two conditions of atmosphere were involved in this study: (1) the puffed wheat was exposed to the air in the tube and (2) the combustion boat containing the puffed wheat was covered by a piece of aluminum foil which was crimped down around the sides of the boat. The heating-microscope study showed that a nitrogen atmosphere did not prevent shrinkage and that it had no effect under a fast heating rate but that it increased the temperature at which shrinkage began when a slower heating rate was used. A few trials in the tube furnace showed that a nitrogen atmosphere had little effect on the carbonizing of the wheat; it was not further investigated. Test firings were made at various temperatures and for various time intervals, both under oxidizing conditions and with the contact of air and wheat restricted by the cover over the boat. The degree of carbonization of the product of each firing was observed. The firing conditions and observations are shown in Table 1.

TABLE 1

Tube Furnace Tests

Temperature		Retention Time	Covered		Observations
°C	°F	(min)	Yes	No	
200	390	15		x	moderately under-fired
		20		x	moderately under-fired
		30		x	moderately under-fired
		30	x		much under-fired
		60		x	slightly under-fired
		60	x		moderately under-fired
230	450	5		x	unchanged
		10		x	much under-fired
		10	x		slightly under-fired
		15		x	slightly under-fired
		20		x	slightly under-fired
		20	x		slightly under-fired
		25		x	slightly under-fired
		30		x	slightly under-fired
		30	x		slightly under-fired
		45		x	black
		45	x		black
245	475	10		x	moderately under-fired
		10	x		much under-fired
		15		x	moderately under-fired
		15	x		moderately under-fired
		20		x	slightly under-fired
		20	x		slightly under-fired
		30		x	slightly under-fired
		30	x		very slightly under-fired
		40		x	black
		40	x		black
260	500	5		x	moderately under-fired
		10		x	black
		10	x		moderately under-fired
		15		x	black
		15	x		slightly under-fired
		20	x		black

DUBURQUE FURNACE TESTS

This electrically heated furnace was 4 by 4 by 9 in., inside. Charges of 3 3/4 cu in. of puffed wheat were put singly into the furnace at temperatures between 220° and 250°C (430° and 480°F) for various retention times. Two different trays made from aluminum sheet were used to hold the charge; one was 2 1/2 by 3 by 1/2 in. deep, and the other 2 1/2 by 3 by 1 in. deep. A charge completely filled the former and one-half filled the latter. Each charge was enclosed by an aluminum cover that fitted the top of its tray. Figure 6 shows the puffed wheat before firing (A) and after firing (B). The weight and volume of each charge was measured before and after firing. Weighings were made on a balance accurate to one decimal and volumes were determined by 100-ml graduate to the closest millilitre. The raw and fired densities and losses in weight and volume during firing were calculated, and the degree of carbonization was observed. Firing conditions and results are shown in Table 2.

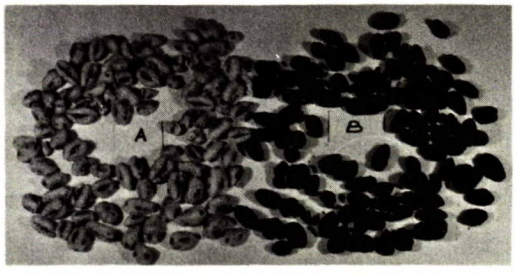


Figure 6: Raw and carbonized puffed wheat.

TABLE 2

Dubuque Furnace Tests

Temperature °C	Temperature °F	Retention Time	Depth (in.)	Raw Density (lb/cu ft)	Fired Density (lb/cu ft)	Weight Loss (%)	Volume Loss (%)	Observations
220	430	2 hr	1/2	2.4	2.4	13	16	considerably under-fired
		2 1/2 "	1/2	3.2	3.0	18	14	ditto
235	455	1 1/2 "	1/2	2.6	2.3	30	19	slightly under-fired
		1 3/4 "	1/2	2.8	2.3	38	26	black
		2 "	1/2	2.7	2.5	47	43	black
		1 1/2 "	1/2 in 1	2.8	2.5	24	14	considerably under-fired
		2 "	1/2 in 1	2.7	2.4	30	20	slightly under-fired
		2 1/2 "	1/2 in 1	2.5	2.2	42	33	black
240	465	3/4 "	1/2	2.3	2.1	39	32	slightly under-fired
		1 "	1/2	2.4	2.1	39	34	black
		1 1/4 "	1/2	2.5	2.2	37	28	black
		1 1/2 "	1/2	2.5	2.1	42	31	black
		3/4 "	1/2 in 1	2.4	2.2	20	18	slightly under-fired
		1 "	1/2 in 1	2.4	2.1	28	17	slightly under-fired
		1 1/4 "	1/2 in 1	2.3	2.0	38	31	black
		1 1/2 "	1/2 in 1	2.4	2.0	39	30	black
250	480	5 min	1/2	2.7	2.6	8	6	no change
		10 min	1/2	2.6	2.1	8	20	considerably under-fired
		15 min	1/2	2.5	3.1	19	30	slightly under-fired
		17 min	1/2	2.4	2.8	23	34	slightly under-fired
		20 min	1/2	2.4	2.8	31	35	black
		22 min	1/2	2.3	2.6	31	36	black
		25 min	1/2	2.7	2.4	32	38	black
		15 min	1/2 in 1	2.5	2.7	11	17	slightly under-fired
		20 min	1/2 in 1	2.3	2.9	17	32	slightly under-fired
		25 min	1/2 in 1	2.3	2.6	25	33	black

DISCUSSION OF RESULTS

The thermal gravimetric analysis (TGA) showed the wheat to be composed of over 85 per cent combustibles. The Dubuque furnace tests indicate that the volatile fraction is about 40 per cent of the total weight, and the TGA showed that this fraction would be driven off below 300°C (570°F).

When puffed wheat kernels were heated in an air atmosphere in the heating microscope, shrinkage began at about 225°C (435°F) under slow heating and at about 265°C (510°F) under rapid heating. While the furnace was flooded with nitrogen, shrinkage of the fragments of puffed wheat began at about 280°C (535°F), if heated slowly, and at about 265°C (510°F), if heated rapidly. These tests show that the puffed wheat contracts under heat, even when oxygen is not present. This indicates that the shrinkage is due principally to the contraction of the kernels but, in an air atmosphere, may be accompanied by some oxidation of carbon. Shrinkage began at about 265°C (510°F) if heating was rapid in both oxidizing and neutral atmospheres. With slow heating, shrinkage began at a lower temperature in an oxidizing atmosphere and at a higher temperature in a neutral atmosphere. In all cases contraction of the specimen began before it had been completely carbonized.

The firing tests in the tube furnace showed that the wheat could be completely carbonized at temperatures between 230° and 260°C (450 and 500°F). The products were carbonized when fired at 230°C for 45 min, at 245°C for 40 min, and at 260°C for 15 min, if exposed to the air, and for 20 min, if covered. In about 55 per cent of the tests, the degree of carbonization was affected less by covering the specimens than by leaving them exposed to the atmosphere in the tube. In another 40 per cent of the tests the carbonization was more advanced with uncovered wheat than with covered wheat. The one instance where the reverse was true (230°C for 10 min) could have been caused by fluctuation of temperature which was controlled manually. These tests were not concerned that some of the products may have been over-fired.

All products that were completely carbonized were reported as "black".

The Dubuque furnace tests were made with two conditions, a 1/2-in. bed of puffed wheat in a 1/2-in. deep tray and in a 1-in. deep tray. Using the 1/2-in. tray, complete carbonization was accomplished at 235°C for 1 3/4 hr, 240°C for 1 hr, and 250°C for 20 min. In the 1-in. tray, carbonization was complete at; 235°C for 2 1/2 hr, 240°C for 1 1/4 hr, and 250°C for 25 min. The lowest fired densities of 2.0 to 2.2 lb/cu ft were achieved at 240°C. An increase of 10 or a decrease of 5 Celsius degrees resulted in higher fired densities. In all tests, there was shrinkage of the kernels; if the product had been completely carbonized, it was at least 30 per cent. The loss in weight in those tests varied between 25 and 47 per cent.

SUMMARY

A single layer of puffed wheat can be carbonized at temperatures between 230° and 260°C (450° and 500°F). The time required for carbonization was between 45 min at the lower temperature and 15 to 20 min at the the higher temperature. A 1/2-in. bed of wheat can be carbonized within the temperature range of 235° to 250°C (455° to 480°F). If the air was restricted to a minimum, the time required varied from 1 3/4 hr to 20 min. When the quantity of air was increased by a volume equal to the bed of wheat, the times required were between 2 1/2 hr and 25 min.

The densities of the carbonized products were between 2.0 and 2.8 lb/cu ft. Shrinkage of the kernels occurred under all firing conditions, varying from 31 to 47 per cent for fully carbonized products. An inert atmosphere (nitrogen) did not appreciably affect the carbonization, although tests in a heating microscope demonstrated that, under slow heating, shrinkage began at a higher temperature in a nitrogen than in an air atmosphere.

Optimum results were obtained with a 1/2-in. bed at 240°C (465°F) and a retention time between 1 and 1 1/2 hr. The time-temperature relationship is critical but, by utilizing the lower end of the carbonizing temperature range, more latitude in the retention time can be gained.

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