CHANGES IN QUALITY OF WHEAT DURING 18 YEARS STORAGE

BY S.W. PIXTON

1. INTRODUCTION

Rapid deterioration in the quality of dry grain is usually caused by insect, mite or rodent attack but even in the absence of these pests quality declines slowly in storage. This investigation was started in 1957 to examine the changes that take place when grain is held in large quantities for a long period in the British Isles. It was assumed that grain deliberately kept on a national scale for several years would be stored in specially constructed containers that could be made airtight or be maintained at a low temperature and that the grain would be pest free. In such containers the grain would be protected from insect and rodent attack and if initially too dry to be vulnerable to fungi would remain so.

Development of fungi and mites can be minimised by keeping the equilibrium relative humidity (e.r.h.) of the grain below 65% but many species of stored product insects can do considerable damage to even drier grain. Whilst all thrive at 70% e.r.h., many can develop at 40% e.r.h. Control of insects is usually achieved by fumigation or with insecticides. Not only are such treatments expensive but they may need to be repeated if the grain remains long in storage and so give rise to objectionable residues or undesirable chemical changes in the grain. Insect development, however, is restricted by low temperature and, in well made storage structures, by the depletion of oxygen. Bailey (1955, 1956, 1957, 1965) showed that certain insects common in storage are killed when the oxygen concentration falls below 2% by volume and concluded that this depletion of oxygen rather than the increase in carbon dioxide was the important factor controlling insect populations. Spratt (1975), however, demonstrated not only that oxygen is used up more quickly when carbon dioxide is present but that adults die at higher oxygen concentrations. Thus more effective control can be expected when the reduction of oxygen is accompanied by a corresponding increase in carbon dioxide. Burges and Burrell (1964) showed that in dry grain at a temperature of 5°C those insects and mites that are not killed are inactive and cause no heating.

Although it is difficult to remove oxygen completely it is commercially practicable to keep concentration low in carefully built storage structures. Also if there is sufficient advantage in storing grain at low temperature economical methods for doing this could be developed. Handling charges form an expensive fixed charge to which storage for long periods, that requires little, therefore adds little. The real cost of long term safety, therefore, is the extra cost of cooling or keeping a low oxygen concentration.

This experiment was set up to investigate the effects of coolness and low oxygen concentration, singly and in combination, on the keeping quality of dry wheat stored for human food and for seed. The experimental design, the wheat characteristics examined, and the results obtained after eighteen years storage are described in this paper.

2. MATERIALS AND METHODS

2.1 Type of wheat used

Two very different types of wheat were stored, a hard Canadian breadmaking wheat No 1 Northern Manitoba, and a soft English wheat, Cappelle, which was suitable for the production of biscuit flour. In 1957 when this experiment was started these were the types of wheats usually stored in the U.K. The Canadian wheat, of the 1956 harvest, had a moisture content of 11.9% on receipt but the Cappelle wheat, harvested in 1957, received at approximately 15%, had to be dried and was eventually used at 12.6%. Both wheats were thoroughly cleaned and aspirated before loading.

2.2 Storage conditions

The wheats were stored in four welded steel cylindrical bins approximately 3 m high and 2 m diameter. These were almost airtight but no attempt was made to ensure perfect sealing. Each bin was divided by wood and hardboard into two semi-cylindrical chambers each of one tonne capacity. One lot of each of Canadian and of English wheat was put separately into each bin (see Fig. 1). To prevent the development of moisture gradients a gentle circulation of the atmosphere in each bin was provided.

Two bins were provided with a continuous supply of fresh air pumped through at a rate equivalent to one complete change of atmosphere approximately every eight days. Before it reached the bins the air was passed through a column of dry wheat (initially at 9% moisture content) which was changed periodically. This arrangement ensured that the moisture contents of the wheats were not much changed and that there was no metabolic loss of oxygen.

In the other two bins the oxygen concentration was reduced to 1%-2% by volume by flushing with nitrogen. The oxygen concentration was measured at intervals and maintained at the correct level by further flushing as required. A slight positive pressure was maintained in the bins to prevent oxygen entering the bins from the atmosphere.

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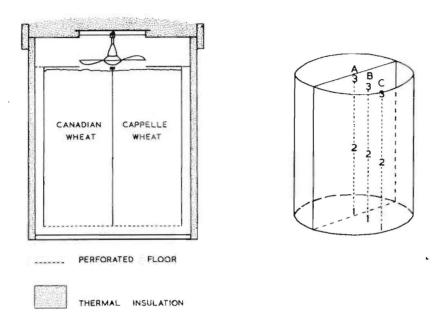


Fig. 1. Section of a storage bin

Fig.2. Sampling points within each bin

A cold room maintained continuously at $4.5^{\circ}C \stackrel{+}{-} 0.5^{\circ}C$ was built to enclose two of the bins one with low oxygen atmosphere and one with normal oxygen atmosphere. The other two bins were exposed to outdoor ambient temperatures, but were thoroughly insulated to simulate conditions within large bulks. All four bins were under the cover of a Dutch barn walled in on three sides.

Temperatures in the centre and at the outer edge of each of the eight wheat bulks, of the cold room and of the ambient air were measured by thermocouples connected to a slow chart recorder. Provision was also made in each bin for gas exchange and for gas sampling.

The four storage conditions are summarised in Table 1.

TABLE 1

Summary of the storage conditions used

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Oxygen concentration<br/>by volumeTemperature<br/>of grain< 2%<br/>Atmospheric4.5^{\circ}C + 0.5^{\circ}C< 2%<br/>AtmosphericAmbient<br/>(Annual cycle<br/>2^{\circ}C - 20^{\circ}C)
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2.3 Tests carried out on the wheats

In order to be able to detect changes, some perhaps very small, which might take place over a long period it was necessary to characterise the wheats in their original condition as closely as possible, and to use for this purpose tests which could be precisely repeated from recorded description alone. The following determinations were made on each type of wheat initially and at intervals of two years.

Moisture content: oven drying, 113°C. 4 hr. (Oxley et al, 1960)

Protein: a) N x 5.7 (Kjeldhal), 13.5% moisture basis

b) N soluble in K₂SO₄, 13.5% moisture basis

Fat: a) Total, by acid hydrolysis, % dry weight basis

b) Free fatty acid, mg KOH/100g grain, dry weight basis Carbohydrate: a) Non reducing sugars as sucrose, mg/10g grain dry wieght basis

b) Reducing sugars as maltose, mg/lOg grain dry weight basisMilling: a) Extraction, % total products

b) Extensibility

b) Colour grade number

Dough tests a) Resiliency

-))__ by extensometer
- c) Water absorption, 1/Kg

Baking tests: pup loaves with 0, 10, 20, 30 ppm potassium bromate added Amylograph maximum viscosity

Titratable acidity of flour, % KH2PO4

Vitamin B, assay

Viability: a) Germinative energy

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b) Germinative capacity, %
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Microflora: a brief examination for living micro_organisms, as described later.

2.4 Sampling

An average sample of each variety of wheat was taken at the start of the experiment by bulking 200 g samples from each 50 Kg of wheat as it was loaded

into the bins in rotation. Subsequently at 2-yearly intervals 1 Kg samples were drawn from each kind of wheat in each bin from nine sampling positions (Fig. 2) by using a vacuum spear. The moisture content of each sample was determined separately and then all other tests listed were made on the thoroughly mixed bulked samples for each wheat from each bin.

2.5 Microflora

A large number of small samples of both types of wheat were collected as the bins were being filled. These were bulked and divided into 10 g portions which were placed in sterile test-tubes. Some of these sub-samples were examined immediately for micro-organisms and the rest distributed randomly between the four bins where they were pushed into the surface of the wheat and so encountered the same environment as the grain bulks. The examination procedure was to culture from a number of whole grains fromeach sub-sample and to make dilution plate counts of fungi and bacteria. A detailed account of the methods used and of the results obtained after 10 yr storage is given by Pixton et al, (1975).

The main experiment was terminated after 18 years when the bins were required for another experiment.

3 RESULTS AND DISCUSSION

3.1 General observations on the wheats

The wheats were examined for signs of mould and for abnormal smell or taste. None was ever detected; the wheats were always bright and free running. 3.2 <u>Chemical analyses</u> (Fig.3)

The content of crude protein, initially 12.9% for Canadian and 8.9% for English wheat, and of salt-soluble protein, initially 2.4% for Canadian and 1.9% for English wheat, remained unchanged under all conditions of storage. Values for total fat, initially 2.9% and 2.4% for Canadian and English wheat respectively, increased by about 0.5%. This small increase can be attributed to metabolism of carbohydrate during the 18 yr storage period.

Free fatty acid values increased steadily in both types of wheat. The rate of increase in the warmer bins was approximately twice that in the cooler bins.

There was a slight reduction in total sugars after 8 yrs storage, then little change of either maltose or sucrose for 8 yrs, then a reduction of both in the next 2 yrs especially in the sucrose. Since both types of wheat, in all four bins, were similarly affected this must be a genuine effect of ageing.

The titratable acidity of the individual flours milled from the wheats (not given in Fig.3), expressed as per cent KH_2PO_4 (Kent-Jones and Amos, 1957) after 18 yrs storage ranged from 0.38% to 0.4% for Canadian flour and from 0.27% to 0.31% for English flour. A figure of about 0.40% is "normal" for freshly milled flour. At 0.60% and above marked deterioration of the baking quality occurs.

| YEARS | | 0 | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 |
|--|------------------------------|----|------------------|---------------------------|----------------|------------------------|---------------------------------------|--------------------|---------------------------|---------------------|----------------|
| mg per 10 g grain dry wt basis | 260 220 180 140 | ð | | a • ⁶ a • • | * . • • • • | 2°0 ,* 8 1214 | * * * * * * * * * * * * * * * * * * * | 0 0 0 0 + + + + | 9 4 0 0 • • • • • • | a g 0 a + * • | ° ° ° ° ° |
| MALTOSE mg per 10g grain dry wt basis | 50 - 30 - 10 - | • | • * * * | **** | : | •••• | ••• | \$ \$ \$ \$ | ÷ | • 2 * • | : * ; ? |
| TOTAL FAT % dry wt basis | 3-2- | ** | 8 * * * * | å å å ÷ | 9 ° • • | ::*: | 0 = 0 0 | ÷ • • • • | 9 <u>.</u> | \$ \$ \$ \$ | * 3 5 6 |
| FREE FATTY ACID mg KOH per 100 g grain dry wt basis | 40 - 30 - 20 - 10 - | ð | | • • • • | •• | 2 a | ** ** | ° s | °.° | 2 * 2 | •• |
| SOL.PROTEIN X I-5% moisture basi | 2·5 - s ^{2·0 -} | * | **** | * . * . | **** | **** | **** | **** | **** | **** | • • • • • |
| U-5 % moisture basis | 9- | ٥ | 0 0 0 0 | 0 8 0 0 | | | 0 0 0 0 | 0 0 0 0 | 0000 | • • • • | 0000 |
| CRUDE PROTEIN 7 | | • | * * • • | | * * * * | * • • • | **** | **** | | | |

Fig. 3 Results of chemical analyses obtained at 2-yearly intervals up to a total storage period of 18 years.

+ Canadian wheat: O English wheat: ● result the same for both varieties. The vitamin B₁ content remained unchanged throughout ranging from 3.6 to
3.7 µg/g (1.15 - 1.18 i.u./g) for the Canadian wheat and from 3.7 to 4.0 µg/g
(1.18 - 1.28 i.u./g) for the English wheat.

3.3 Milling and physical dough tests (Fig.4)

The wheats were milled on a Buhler mill with constant setting, flour yield being calculated as a percentage of total products. This procedure takes into account moisture and product loss in milling and enabled a comparison to be made of yields and colour numbers at different sampling times. The colour number gives an indication of the contamination of the flour by fine bran particles. The lower the colour number the whiter the flour and the lower is the ash content. A low number would be obtained if the endosperm separated easily from the bran during milling which would also lead to a higher yield. Starch cells becoming more friable with age would have the same effect.

Over the total storage period yields varied irregularly with the highest yields in the last two years of storage. There was no correlation between flour yield and colour number, but it is interesting to note that the lowest numbers were obtained in the last four years of storage when the yields were the highest.

Water absorption, which is the amount of water a dough will take and hold during fermentation, remained fairly constant, for both types of wheat, throughout the total storage period at about 0.55 1/Kg for Canadian wheat and

| YEARS | | 0 | | 2 | | | 4 | | | 6 | | | 8 | | - | 10 | | - | 12 | | | 14 | | | 16 | | | 18 | 5 |
|------------------|---------|---|---|-----|-----|---|---|-----|---|---|------|----|---|-----|----|------|-----|---|-----|-----|-----|-----|-----|---|-----|----|---|------|--------|
| BIN | | | 1 | 2 3 | 4 | 1 | | 5 4 | 1 | | 1 4 | 1 | | 3 4 | | | | 1 | 2 3 | | | | 4 | | 2.3 | 4 | | 2) | |
| | 10 | | 1 | | | | | | | | | | | | | | | | | | | | | | | ۰ | | 4 | 3 |
| 7. | 40 | | | | | | | | | | | | | | | | | | | | | | 4 | | | • | | | |
| GERMINATION % | 1 | | | | | | | | | | | | | | | | | | | • | | | | | 9 | | | | - |
| | 70 | | | | | | | | | | | | | | | | .9 | 1 | | * | | | | | | | | 1 | r |
| | 100 | | • | | | | • | • • | | • | | | | • . | | • . | | | | | | • . | | | | | | • | |
| CONTENT % | 12 - | * | | • • | | • | • | •• | + | • | •• | + | • | + + | | • • | • • | • | • • | + | | • • | | | • . | | | | |
| MOISTURE | 13- | - | | | | • | | | | 0 | | 0 | | 0 a | 0 | • • | | 9 | ۰, | | | a . | . a | 0 | • • | | | • | |
| EXTENSIBILITY | 10- | 0 | • | ٥. | | 0 | • | | • | a | • • | | ٩ | 0.0 | 4 | • | | • | • } | | å | • | . 9 | | • • | * | | • | 2 |
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| SPRING | 600 | | | | | ā | | ** | | • | 4 | | • | • • | 8 | ŧ. ; | 5 8 | | | | + 0 | | | | | • | | : ; | |
| I/Kg | .4 - | 9 | 0 | 3.4 | | | _ | | l | 0 | | | | 0.0 | | 0 4 | 0 | • | 8 | | a | 0 0 | | | • | | | a 1 | P |
| WATER | | * | * | * | • • | * | * | ••• | | • | • • | | • | • • | 1: | • | • * | * | • | 1 | | • ' | • • | | * * | • | | • • | • |
| GRADE NO. | 0 - 0 - | - | - | | _ | | _ | | - | | | - | | | - | | _ | 1 | _ | _ | 1 | _ | | - | * | _ | | * | _ |
| COLOUR | 4 | 2 | 8 | • • | e. | | 7 | | 2 | ę | : | \$ | å | •• | • | • | 2 e | • | | * * | \$ | | . 1 | 8 | | | • | • 1 | 8 |
| VIELD %. | 65- | - | - | - | - | ę | - | • • | 1 | | | - | _ | _ | - | | _ | - | _ | _ | | + | | - | _ | _ | | _ | - |
| FLOUR | 75- | • | * | * | : | | | ŧ. | 8 | • | • • | 5 | 6 | å å | • | • • | ė : | • | | 2 ° | • | 0 | : : | • | - | \$ | 4 | \$ 1 | 2 |

Fig.4. Results of flour yield, colour number, physical dough tests, moisture content determination and germination tests obtained at 2-yearly intervals up to a total storage period of 18 yrs. + Canadian wheat: O English wheat;
result the same for both varieties.

Spring (resiliency), as the name implies, measures the elastic properties of the gluten, i.e., its ability to recover after stretching. Extensibility measures the capacity of the dough to stretch without breaking. As the storage period increased the dough from both the Canadian and English wheats became "shorter" in that spring increased and correspondingly extensibility decreased. This is a well known ageing phenomenon and was more marked in the wheats stored in the warmer conditions especially those in normal air. However in the last two years spring showed a tendency to decrease indicating that at the end of the storage period the gluten was softening, presumably because of enzymic action.

The amylograph maximum viscosity (not given in Fig.4) gives a measure of the activity of the enzyme amylase in the flour which is essential to the baking process in the production of carbon dioxide in fermentation. The maximum viscosity, particularly for the Canadian wheats tended to increase with time. For both types of wheat the viscosity was higher in the warmer storage conditions. This first became apparent after 10 yrs storage, at about the same time that a decrease in total sugars occurred. We do not know if the amylase activity decreases or if, during storage, the starch becomes less susceptible to amylase attack.

3.4 Baking tests

Throughout the storage period the flour milled from the Canadian wheat responded to the addition of potassium bromate, the optimum response being obtained with a treatment rate of 20 ppm. The "bromate response" is the amount of potassium bromate required to obtain optimum improvement in a dough. Generally, the higher the response the stronger is the flour. The volume of a loaf made with Canadian flour with 20 ppm potassium bromate added was initially 715 cm³ when the wheat was first stored. After the wheat had been stored at low temperature for 18 yrs the loaf volume was 815 cm^3 and if storage was at ambient temperature the volume was 760 cm³. Over the last two years of storage there was a decrease in loaf volume. This supports the contention made earlier that the gluten was softening. English wheat, primarily a biscuit wheat had very little bromate response, initially the loaf volume being 575 cm³ rising to 640 cm³ at the end of storage.

In the baking process the quantity of carbon dioxide produced during fermentation depends on the level of alpha amylase and damaged starch cells present in the flour as well as on the level of reducing sugars. Fungal alpha amylase was added to both the Canadian and English wheat flours, at the rate of 224 ppm, to correct a deficiency of natural amylase which had decreased after the first 8 yrs of storage. Good loaves were always obtained from the Canadian wheats those stored at low temperature producing marginally the best loaves.

3.5 Moisture content (Fig. 4)

The overall mean moisture content of both types of wheat in all four bins rose by about 0.5% during the first two years and thereafter remained virtually constant over the entire storage period. The difference between the moisture content of the two wheats, in the same bin, after several years of storage varied from 1.1% to 1.3%. This is an expression of the different moisture content/ $e_*r_*h_*$ relationship of the two wheats.

Throughout the period of storage the moisture content of both types of wheat at low temperature tended to be higher at the top and side than the bottom and centre of the bulk. By contrast, in the warmer bins the moisture content at the bottom and centre tended to be higher than the side and top and the differences were slightly greater. These slight vertical and horizontal gradients were probably caused by temperature and consequent vapour pressure gradients that accompany seasonal changes of climate.

3.6 Viability (Fig. 4)

Under ambient conditions the germinative capacity fell during storage. The Canadian wheat began to lose both energy and capacity gradually between 10 and 12 yrs and more steeply between 14 and 18 yrs when it had declined to 4%. This process started two years earlier in the English wheat which fell to zero germination after 18 yrs storage. At low oxygen tension the loss in capacity started a little later, after about 12 yrs storage. It was the more marked in the English wheat in which the capacity fell to 19% after 18 yrs compared with 77% for the Canadian wheat.

At low temperature, however, the germinative energy and capacity of the wheats were maintained over the entire 18 yrs of storage in the bins. For this reason about 50 Kg of the wheats stored at low temperature and normal oxygen tension were kept in a closed metal container in a room at 5° C, and now, after 22 yrs, the germinative capacity for the Canadian wheat is still 95% and that of the English wheat 96%.

3.7 Microflora

No growth of micro-organisms took place in samples of the wheats stored up to 10 yrs in tubes, dryness being the main controlling factor. However, many micro-organisms were viable on the wheats at the end of this period. Low oxygen tension had little or no effect on the survival of micro-organisms but, with the exception of <u>Aspergillus</u> that survived almost as well at ambient temperature as at low temperature, all diminished much more quickly at ambient temperature. This decline in fungal viability paralleled the decline in grain viability which was also greater under ambient conditions. If there was a slight increase in the moisture content of the wheats, growth of xerophilic fungi such as the <u>Aspergillus restrictus</u> and <u>A. glaucus</u> groups, could take place at ambient temperature. A greater increase in moisture content would be necessary before fungal growth could take place in the low temperature conditions.

4. CONCLUSIONS

No change has taken place in the germinative energy or capacity of the wheats stored at 5° C for 22 yrs. Both varieties of wheat stored at ambient temperature and oxygen tension have little or no viability after 18 yrs storage. Low oxygen tension in the ambient temperature conditions appears to have supported viability for a longer period, but both wheats showed a reduced energy.

Free fatty acid values throughout have continued to show an increase. The rate of increase, and the final level of free fatty acid of the wheats in the warmer bins was about twice that found in the cold bins. In the last two years both reducing and non-reducing sugars have decreased again in both types of wheat in all bins.

Whilst water absorption has remained steady, resiliency increased with time at the expense of extensibility, but, in the last two years of storage resiliency of the Canadian doughs decreased indicating that the gluten is softening possibly due to enzymic action. This is also reflected by a fall in loaf volume.

Although moisture changes were unimportant in this experiment with dry grain, they could be serious in grain with a higher initial moisture content or where

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there are violent climatic fluctuations.

With the exception of <u>Aspergillus</u> which survived almost as well at ambient temperature as at low temperature all micro-organisms diminished much more quickly at ambient temperature. There was no development of fungi, dryness being the controlling factor.

Clean dry wheat, providing it is protected from atmospheric moisture, from rapid changes of temperature and from insect infestation will still produce good loaves after 18 yrs storage in spite of the fact that germinative energy and capacity are greatly reduced. It is necessary, however, to add alpha amylase to the flour in the baking process to compensate for loss of the natural enzyme. Low temperature prevents a decline in viability but otherwise has only a marginal effect on loaf quality. Low oxygen tension, whilst giving some support to viability at ambient temperature, had no effect on loaf quality.

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