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ON THE BLEACHING OF FLOUR AND THE ADDITION OF SO-CALLED "IMPROVERS" TO FLOUR.

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(*Abstract of a Report to the Local Government Board, England. Food Reports No.* 12.)

1.—BLEACHING PROCESSES.

THE practice of flour bleaching originated in this country about ten years ago; bleaching, however, is said to have been attempted in France two or three years prior to this. The practice was rapidly adopted by millers in the United Kingdom, in America, and elsewhere. Such bleaching is applied only to roller-milled flour. Numerous patents have been granted for bleaching appliances and for the application of various chemicals to the bleaching of flour. Ozone, chlorine, oxides of chlorine, bromine, nitrogen peroxide and other oxidising substances have been suggested and tried as bleaching agents. Of these substances nitrogen peroxide is the only one which has given satisfactory results, and all commercial bleaching of flour at the present time depends on the use of this substance. It can be produced in various ways and from a variety of materials. Two methods of production, which may conveniently be termed the chemical and electrical methods, are in use commercially at the present time. In the first of these nitrogen peroxide is produced by the action of nitric acid on ferrous sulphate, the amount of gas formed being controlled by regulating the supply of acid and ferrous sulphate. A current of air charged with the gas is led through a revolving reel or "agitator," as it is called, through which a stream of flour is continually flowing. The flour in its passage is thoroughly exposed to the action of the nitrogen peroxide, and is bleached when it emerges from the agitator. In actual practice this process possesses certain commercial disadvantages. The apparatus requires constant attention if the degree of bleaching is to be properly controlled; overbleaching and condensation of acid resulting in local staining of the flour are liable to occur, while in the hands of ordinary workmen the use of strong acids is not entirely free from risk. As regards expense, it compares unfavourably with other methods, some of which are said to be worked at one-tenth of the cost of the acid process.

Another chemical process depends on the production of nitrogen peroxide from the combustion of ammonia in air. A current of air, mixed with ammonia gas obtained from an iron cylinder containing liquid ammonia, is pumped through a narrow platinum tube. The tube becomes red hot, and the oxygen of the air and the ammonia interact with the production of nitrogen peroxide. The air charged with nitrogen peroxide is passed together with flour through an agitator in the way already described. The apparatus is compact, and the process is free from the drawbacks attendant on the use of nitric acid and ferrous sulphate. It is suitable for mills where the output of flour is small. Nitrosyl chloride has also been proposed as a source of nitrogen peroxide.

The electrical methods of bleaching depend upon the fact that an electrical sparking discharge in air results in the combination of a certain amount of the nitrogen and oxygen of the air to form nitrogen peroxide. Electrical methods of bleaching are rapidly superseding chemical processes on account of their simplicity in operation and the greater accuracy with which the degree of bleaching can be controlled and overbleaching avoided. Condensation of acid, resulting in local staining of the flour, is said to be less likely to occur than in the chemical processes. Condensation is said to be prevented if the room in which the agitators are placed is kept warm.

In one form of electrical apparatus two metallic electrodes connected with a suitable source of current, generally a small dynamo, are alternately brought into contact and separated, an intermittent flame-discharge occurring at each separation of the electrodes. Air is pumped over the electrodes, and at each flash becomes charged with nitrogen

https://doi.org/10.1017/S0022172400016612 Published online by Cambridge University Press

peroxide. The concentration of nitrogen peroxide in the air can be regulated, within limits, by altering the amount of air pumped over the electrodes. In another form of apparatus, electricity supplied from a dynamo is transformed to a potential of 10,000 volts or more and conveyed by well-insulated conductors to fixed metallic electrodes (generally four pairs) between which a continuous sparking discharge occurs. In another part of the apparatus provision is made for the production of a brush discharge, and a current of air is pumped first over this and then over the sparking electrodes. In its passage through the brush discharge, part of the oxygen of the air is ozonised, and in its passage over the electrodes nitrogen peroxide is produced. The air, charged with nitrogen peroxide and ozone, is brought into contact with flour in an agitator in the usual way. It is now generally believed that ozone has no bleaching action on flour, and although originally an electrical bleaching machine was constructed with a view to the production of ozone, in the belief that this gas was the active bleaching agent, any bleaching which occurred was probably due to nitrogen peroxide formed simultaneously with the ozone. It may here be noted that the introduction of electrical methods of bleaching has been accompanied by extensive litigation, dealing with patents and patent rights[1].

In these electrical bleaching machines the degree of bleaching obtainable can be varied either by altering the amount of air passing through the apparatus or by varying the strength and frequency of the electrical discharges. Quite recently a form of apparatus has been introduced which, it is claimed, enables the bleaching process to be controlled with greater precision than in any of the foregoing processes. In this apparatus a current of air under pressure is blown from a fine jet through an electric arc formed between two brass electrodes. A flame is thus produced the size of which, and, consequently, the amount of nitrogen peroxide generated, can be regulated with some accuracy by varying the strength of the current supplied to the machine[2].

Millers are guided entirely by the appearance of the issuing flour in regulating and controlling the bleaching apparatus; they have no

[1] Flour Oxidising Company, Ltd. *v.* J. & R. Hutchinson (*Times*, 28th April, 1909).

[2] Commonly a current of 7 to 10 ampères at 100 volts is transformed to about 14,000 volts, at which potential the discharge occurs. Two to five cubic feet of air per minute, according to the pressure (generally about five inches of mercury), pass through the apparatus, which is capable of bleaching two to three tons of flour per hour. The inventor was, however, unable to give me any information as to the amount of nitrogen peroxide which the apparatus contributed to the air.

https://doi.org/10.1017/S0022172400016612 Published online by Cambridge University Press

knowledge of the actual amount of nitrogen peroxide produced or used. Examination of the bleaching gas at different mills was made in course of my inquiry, and it was found that very different degrees of concentration of nitrogen peroxide were employed.

2.—CONSIDERATIONS INFLUENCING THE AMOUNT AND DEGREE OF BLEACHING.

The practice of millers as to the bleaching of flour varies with the circumstances of their trade. In Ireland, a demand exists for a very white flour; a taste for such flour probably arose on account of the importation of American flour, which "ages" naturally during transit, and in this way becomes extremely white. For scone-making and for household purposes generally, a white flour is also desired; it is stated that the yellow tinge which baking powders containing soda in excess may give to scones is rendered less apparent by the use of a very white flour. Millers, therefore, who supply places where for these or other reasons there is a particular and constant demand for very white flour, naturally find bleaching of special service. Apart from this, it has to be remembered that it may be cheaper for the miller to utilise wheats which normally would yield a comparatively dark flour. The number of such wheats now available for the miller is large, and some of them yield flour which possesses excellent baking qualities but is inferior in consequence of the importance which has come to be attached to colour. Among the wheats which yield flour for which on this account bleaching is considered specially useful, are certain Indian wheats, Hard Plate, Walla Walla, durum, and some varieties of Russian wheat. But even the naturally whitest flours may be bleached for districts where there is a demand for excessively white flour.

There are also other ways, to be referred to later, in which bleaching may enhance the value of particular flours to the miller. In these circumstances considerable differences in the practice of bleaching obtain not only in mills in different parts of the country, but frequently, also, in the same mill from day to day.

It must not be supposed that bleaching in this country has yet become universal, even amongst the larger firms. Many millers have never bleached flour for sale. Some of these are at present only deterred from installing bleaching plants by considerations of expense. Others have had bleaching plants installed but have discontinued using them—in some cases for the reason that they were unwilling to pay the patentees the sum demanded for permission to use the process. Other millers who have abandoned bleaching have reconsidered their decision, and have resumed the practice. Bleaching has been carried on uninterruptedly since its adoption by certain millers, who have found it to their advantage commercially.

It appears to be the custom of many millers, when using a bleaching plant, to bleach all the flour produced. This, of course, would necessarily be the case whenever a straight run[1] was being milled and bleached. Some millers state that although they bleach their whole output they make a distinction between the lower and higher grades[2], bleaching the latter more than the former. They believe that the best results from bleaching are obtained with the higher grade flours, and that the bleaching of lower grades, unless carefully done, is liable to mar rather than improve the appearance of the flour. In some mills, where a blend containing, say, Indian wheat is used, a larger proportion of flour from Indian wheat may appear at the bottom ("tail") of the mill, *i.e.* in the lower grades, than at the top of the mill. In this case the lower grades may be subjected to stronger bleaching than the higher grades. In certain cases the miller has found that his customers prefer high grade flour unbleached, and has, in consequence, restricted bleaching to low grades only.

The degree to which the bleaching of flour is carried depends on the kinds of wheat used; on whether the demand to be met is for very white flour or for flour with a slight yellow tinge or "bloom," and on similar considerations. Many millers content themselves with lightening the colour till only a slightly yellow tinge is perceptible—merely "touching" the flour, as they term it. Others are not satisfied with this but bleach until the flour is quite white. Certain millers consider this to be excessive, and assert that the dead whiteness thus obtained is unnatural and unpleasant.

Some millers, *e.g.*, in Glasgow, where baking and milling methods differ somewhat from those in other parts of the country, do not bleach flour from spring wheat but bleach winter wheat flours, although in general these are whiter than those obtained from spring wheat. These firms largely supply bakers who are accustomed to the tinge or

[1] By the term "straight run" is meant the whole of the flour produced in the mill without any separation into different grades.

[2] Here and elsewhere when the text requires it, such terms as "high," "low," "good" or "poor" when applied to grade or quality are used in the trade sense, and not in reference to food values.

"bloom" natural to spring wheat flour, and have come to regard it as characteristic of this class of flour. On the other hand, these bakers prefer winter wheat flour, which is used in the "doughing" stage of baking, to be as white as possible. Winter wheat flour is also used for home baking and scone making, and for these purposes is desired specially white, consequently it is the common practice of millers possessing bleaching plants, and milling spring and winter wheats separately, to bleach only the winter wheat flours as white as possible, and to leave the spring wheat flours untouched.

3.—THE EFFECT OF BLEACHING ON THE APPEARANCE AND OTHER PROPERTIES OF FLOUR.

The precise effect upon flour of bleaching by nitrogen peroxide is a matter of considerable dispute amongst millers, bakers and others who have given attention to this question, and who either favour or oppose the practice. The most obvious effect of nitrogen peroxide is the change of colour which it produces in the flour, but other more subtle changes, such as alterations in the baking qualities of the flour, are also attributed to its use.

The chief opponents of bleaching in the trade are many bakers, flour importers, and those millers who for one reason or another do not bleach. Bakers are not accustomed to combined action in matters of this kind, but in Glasgow, in 1906, the principal bakers issued a circular protesting against the bleaching of flour.

The Effect of Bleaching on Baking Qualities.—A number of investigations have been made on this point, especially in America. In many cases no differences have been observed in respect of the baking qualities between bleached and unbleached flour; in cases where differences have been observed they are small, and may well be within the limits of experimental error, or be due, where the baking qualities appear to be improved, to the slight drying of the flour during the bleaching process, or, where the baking qualities appear to have deteriorated, to excessive bleaching.

Avery(3) was unable to detect any difference in the baking qualities of bleached and unbleached flour, and Alway and Pinckney(2) have confirmed his observations.

Snyder(15) came to the conclusion that bleaching has practically no effect on the baking qualities of a flour. In some of his experiments slightly larger loaves were obtained from bleached flour than from unbleached flour; this he attributed to slight drying of the flour during bleaching.

Ladd and Stallings(10), on the other hand, assert that the gluten of flour deteriorates as a result of bleaching, and the water-absorbing capacity of the flour diminishes, consequently a larger loaf can be produced from unbleached than from bleached flour.

Amongst the bakers and millers (both bleaching and non-bleaching) whom I have visited the opinion is common that ordinary commercial bleaching produces little or no effect on the baking qualities of flour.

Conclusions as to the effect of bleaching upon flour.—It seems probable that bleaching, when carefully carried out, produces no appreciable effect upon the baking qualities of flour.

It may be true that by mere bleaching it is impossible to make the lowest grades of flour equal in appearance to the highest grades, but there can be no doubt that bleaching is capable of improving the colour and therefore the commercial value of the lower grades of flour. This is borne out by the common practice of bleaching the whole output of the mill, both high and low grades; also by statements of manufacturers of bleaching apparatus that bleaching improves both high and low grade flours, and enables the miller to produce "a longer length of patents," and further by the fact that some millers bleach only their lower grades of flour. Bleaching would seem to improve, from the miller's point of view, the colour of the whole output of the mill, although the improvement may be less marked in the lower than in the higher grades which are practically free from offal.

That this improvement represents a pecuniary gain to the miller is evident from the longer patents—*i.e.*, the larger percentage of high grade flour—which can apparently be obtained as a result of bleaching. This advantage must be remembered in connection with the assertion that the price obtainable per sack is not increased by the bleaching process.

It is probable that bleaching is of assistance in maintaining uniformity in the appearance of flour milled from different wheats, but at the same time it enables the miller to use cheaper wheats and to transform what would otherwise be a dark-coloured cheap flour into a whiter, higher-priced article, the gain due to the enhanced price going to the miller. It is said that a saving of 6*d.* to 1*s.* per sack may thus be effected by the miller.

Bakers, importers and others judge flour largely on its appearance and are accustomed to associate whiteness with high commercial quality and with care and attention in the choice and milling of the wheat. Owing to bleaching, colour is no longer a trustworthy criterion, and a purchaser may be supplied with an article of a different quality from that which he believes he is receiving. Millers are, of course, aware of this, and show a disinclination to inform their customers that their flour is bleached.

There seems no sufficient evidence that mere bleaching can counteract defects in flour due to unsoundness in the wheat from which it was milled.

4.—CHEMICAL CHANGES OCCURRING IN FLOUR AS A RESULT OF BLEACHING BY NITROGEN PEROXIDE.

Although very little is known with certainty regarding the chemical changes which occur in flour on treatment with nitrogen peroxide, I may here refer to results which have been obtained, particularly in America, by numerous observers whose investigations have been published.

It has been ascertained that a watery extract of bleached flour reacts to the Griess-Ilosvay test, showing the presence of a nitrite-reacting substance as a result of treating the flour with nitrogen peroxide. This reaction is not given by ordinary unbleached flour when care is taken to prevent its contamination with nitrites from the air or other sources. Warington(17) drew attention to the delicacy of this test as a means of detecting the presence of nitrites, and, later, Willard and Ladd employed it in connection with the examination of flour. Alway and Gortner(1) have described a method of applying this test to flour, and are convinced of its reliability, if the precautions enumerated by them are observed. Weil(18), however, has recently stated that certain flours which have not been bleached respond to the Griess-Ilosvay test, and recommends instead that the flour be exposed to the action of sulphuretted hydrogen for one hour. Unbleached flour is unaltered by this treatment, whereas bleached flour becomes darker and acquires the colour it possessed before bleaching. Shaw(13) has proposed an alternative test, which has as its object the detection of nitro-derivatives in the flour. A concentrated alcoholic extract of bleached flour, treated with a drop of a solution of diphenylamine in sulphuric acid, gives a blue colouration. Alway and Gortner(1) have not found this test reliable in their hands.

The amount of nitrite-reacting substance, which can be demonstrated in bleached flour, varies considerably in different samples. In some cases only traces can be detected; in others as much as 40 parts of nitrite (expressed as sodium nitrite) per million of flour have been found. The results obtained are variously given in parts of nitrite (expressed as sodium nitrite) or in parts of nitrite-nitrogen per million or per ten-million parts of flour.

Nitrates are also formed as a result of bleaching. When bleached flour is made into bread, from one-half to two-thirds of the nitrite present in the bleached flour disappears, and an increase in nitrates is said to occur. In biscuits practically all the nitrite disappears on baking. Alway and Pinckney(2) have found that no relation exists between the amount of nitrite which may remain in bread and that originally present in the flour; sometimes no nitrite may be left in the bread. Snyder(15) has suggested that the nitrite-reacting material in flour is either ammonium nitrite or a volatile compound of similar nature.

From the work of Fleurent(6) and others the bleaching of flour appears to be due to the destruction of yellow colouring matter dissolved in a thin layer of oil which surrounds each granule of starch. Ladd(9) has stated that changes in the appearance and in the composition of the oil, as evidenced by a lowered iodine number and by the introduction of nitrogen into the oil, result from the process of bleaching by nitrogen peroxide. The oil of flour, which had aged naturally for nine months, preserved its iodine number unaltered, whilst the iodine number of oil from the same flour, bleached with nitrogen peroxide and aged for the same length of time, had diminished considerably. Fleurent(6) also found that bleaching decreased the iodine number of flour oil, and states that the whiteness obtained by the action of the nitrogen peroxide on flour is due to the white starch granules becoming more obvious as a result of the decolourisation and increase in transparency of the oil film surrounding them, whereas the whiteness resulting from age is due to conversion of the oil into fixed fatty acids which are white in themselves.

Gill(16) asserts that bleaching in addition to lowering the iodine number converts a portion of the olein into elaidin.

Gustav Mann(16) states that the oil of flour is altered by treatment with nitrogen peroxide in such a way as to make it more difficult to stain with scarlet R or Soudan 3 than oil from unbleached flour.

The acidity of flour is stated by Balland(4) to increase as a result of treatment with nitrogen peroxide, but Alway and Pinckney(2) state that this is not the case if less than 50 c.c. of nitrogen peroxide per kilogram of flour be used.

It has been suggested that the formation of diazo- and nitro-compounds may result from the interaction of nitrogen peroxide and the gluten or protein of the flour. Skraup(14) and others have shown that nitrous acid can attack and profoundly alter the composition of proteins. Gustav Mann(16) states that the amount of nitrite present is no indication of the extent to which the protein may have been attacked, and draws attention to the possibility of the destruction of important amino-groups in the protein molecule as a result of bleaching, whereby its value as a foodstuff would be greatly diminished. Folin(16) concurs with this view, and believes that nitro-compounds may be formed as a result of the action of nitrogen peroxide on the protein of flour.

Other investigators maintain that little if any chemical change in the flour, other than the decolourisation of the oil and the presence of traces of nitrite-reacting material, can be detected as a result of treatment with nitrogen peroxide. Avery(3) has also come to this conclusion, and is of opinion that only in overbleached flour can appreciable changes, such as the diminution of the iodine number, be detected. Snyder(15) has likewise failed to obtain any change in the iodine number as a result of bleaching, and considers that Ladd is not justified in his conclusion that nitrogen is introduced into the flour oil by bleaching, since the oil from wheat flour normally contains a small amount of nitrogenous matter which is not increased by bleaching. He further maintains that in commercial bleaching only sufficient nitrogen peroxide is used to bleach the flour, and that the risk of forming diazo- and nitro-compounds is negligible. He also asserts that practically all the nitrogen peroxide brought into contact with the flour in the commercial process of bleaching can be accounted for, either in the air leaving the agitator or as physically combined (adsorbed) in the flour, and he attributes bleaching to the catalytic action of the nitrogen peroxide, which does not itself chemically combine with any of the constituents of the flour. Wesener and Teller(19) are of the opinion that no change except as regards colour is produced in flour as a result of commercial bleaching. They admit, however, that changes in the composition of the protein and fat may be caused by overbleaching.

The amount of nitrogen peroxide required to produce bleaching has also received attention. Avery(3) states that the nitrogen peroxide produced by 3 c.c. of nitric oxide in 3 litres of air will bleach 1 kilogram of flour; maximum bleaching was attained with 40 c.c. of nitric oxide; with amounts above this the colour of the flour deteriorated. Hale(7) considers that the best results are attained by using 30 c.c. of nitric oxide to 1 kilogram of flour. Fleurent(6) states that according to the kind of flour from 15 to 40 c.c. of nitrogen peroxide is sufficient for 1 kilogram of flour. Snyder(15) found that 5 c.c. of nitrogen peroxide was sufficient to bleach 1 kilogram of flour; and Alway and Gortner(1) state that from 100 to 150 c.c. of nitric oxide are required to produce a maximum bleaching effect.

Shepard(16) points out that millers do not know how much nitrogen peroxide they use, but judge entirely by the colour of the flour, and that, relatively, large amounts (more than 100 parts of nitrogen peroxide per million of flour) may be absorbed and the flour remain a strictly commercial article not overbleached. He considers that in ordinary commercial bleaching 36 to 72 parts of nitrogen peroxide are absorbed by a million parts of flour and that even larger quantities may be required in the case of highly coloured flours.

Various observers have found very diverse amounts of nitrites in flour as a result of laboratory treatment with different amounts of nitrogen peroxide. A few of the values obtained are given in the table below. The figures in brackets represent the amount of nitrogen peroxide in cubic centimetres added to one kilogram of flour; the other figures opposite these represent, approximately, the parts of nitrite (expressed as $NaNO_2$) found in a million parts of flour after treatment with the respective volumes of nitrogen peroxide:—

Alway and Gortner(1).	Hale(7).	Snyder(15).
(5 c.c.) 6 parts.	(10 c.c.) 7 parts.	(5 c.c.) 2 parts.
(25 c.c.) 20 parts.	(20 c.c.) 8 parts.	(25 c.c.) 5 parts.
(50 c.c.) 30 parts.	(50 c.c.) 36 parts.	(50 c.c.) 16 parts.
(100 c.c.) 40–60 parts.	(100 c.c.) 40 parts.	(100 c.c.) 20 parts.

The results of laboratory experiments on the same point will be found in Dr Monier-Williams' report (see *This Journal*, XI. p. 167).

The amount of nitrite-reacting material in bleached flour tends to diminish with lapse of time when a certain degree of bleaching has been exceeded.

Snyder(15) found in one sample, bleached with 250 c.c. of nitric oxide per kilogram of flour, that 24 days after bleaching the amount of nitrite had decreased by one-half, *i.e.*, from 8 parts per million to 4 parts per million; the latter amount can be obtained by 25 c.c. of nitric oxide. Alway and Gortner(1) observed that nitrites in bleached flour rapidly diminish in amount when the flour is kept if more than 50 c.c. of nitric

https://doi.org/10.1017/S0022172400016612 Published online by Cambridge University Press

oxide per kilogram of flour has been used for bleaching. When less than this amount of nitric oxide has been used, no appreciable change in the nitrite content of the flour takes place on keeping.

The amount of nitrite-reacting material present in flour, as indicated by the Griess-Ilosvay test, is not necessarily any criterion of the degree of bleaching or of the amount of nitrogen peroxide which may have been absorbed by the flour. It would appear from experiments by Snyder(15) and by Alway and Gortner(1) that the nitrite content of flour varies with the degree of bleaching only up to a certain point, and beyond this is not permanently increased by further bleaching. The latter investigators have observed an approximate proportionality between the amount of nitric oxide used for the production of nitrogen peroxide and the nitrite produced up to 50 c.c. of nitric oxide per kilogram of flour. Thus, 2 c.c. of nitric oxide produced about 3 to 4 parts of nitrite per million of flour; 5 c.c. about 6 parts per million, 25 c.c. about 20 parts per million, and 50 c.c. about 30 parts per million. Larger amounts than this produce an increase in the nitrite content which is only temporary, and, as already mentioned, rapidly diminishes on keeping. It is, therefore, not justifiable from a mere determination of the nitrites in flour to draw any conclusions as to whether the flour has been subjected to excessive bleaching.

The work done on the chemical changes in flour as a result of bleaching has been incomplete, and the conclusions of the different investigators contradictory in several respects, it was arranged that some of the more important considerations should be investigated experimentally by Dr Monier-Williams (see *This Journal*, XI. p. 167). The results which he has obtained should be studied in connection with the above review. It would appear from his experiments that maximum bleaching is attained with 30 to 100 c.c. of nitrogen peroxide per kilogram of flour. In freshly bleached flour the amount of nitrite is proportional to the nitrogen peroxide used, amounting to about 30 to 40 per cent. of it. In the more highly bleached samples a decrease in nitrite was observed after a lapse of some days, but, practically, no alteration in the nitrite content of slightly bleached samples occurs. It appears that about 60 per cent. of the nitrogen absorbed as nitrogen peroxide can be extracted by water in the form of nitrites and nitrates; the remainder has probably entered into combination with or is absorbed by various elements of the flour. Dr Monier-Williams' work shows clearly that, with higher degrees of bleaching, definite changes are produced in the constitution of the flour. Among these are an increase in soluble proteins and carbohydrates; the oil of the flour acquires the character of an oxidised oil, and about 6 to 7 per cent. of the nitrogen introduced into the flour as nitrogen peroxide is absorbed by the oil. No evidence of the formation of diazo-compounds was forthcoming.

The Value of Chemical Methods in the Detection of Bleached Flour.—The chemical change most easily demonstrated in flour as a result of bleaching is the formation of nitrites. The Griess-Ilosvay reaction when properly carried out affords an exceedingly delicate test for the presence of nitrites, and on this account has been largely used for the detection of nitrites in flour. Flour which gives a positive reaction with the Griess-Ilosvay test is commonly assumed to be bleached, but to infer bleaching from the mere occurrence of the nitrite reaction is in many cases not permissible.

In the first place, owing to the great delicacy of the test, care has to be taken that the materials used for the test, especially the water used for extracting the flour, are perfectly free from all traces of nitrites. Distilled water commonly contains nitrites, but in the majority of cases, tap water is practically free from nitrites, and when boiled over an electric heater to free it from dissolved oxygen, which may affect the test adversely, is suitable for use in the estimation of nitrites. The test itself should be performed in a room in which no flame is burning, since the air may in this way become contaminated with nitrous acid.

Flour readily absorbs nitrous acid from the air, and in manufacturing towns amounts easily appreciable by the Griess-Ilosvay test may gain access to the flour. In Bulletin 206 of the Laboratory of the Inland Revenue Department, Canada, reference is made to a sample of flour originally free from nitrites which was exposed to the air on the laboratory roof; after ten days it was found to contain 1·6 parts per million of nitrite and after 25 days 3 parts per million. In this case, however, the four ventilating flues from the laboratory in which all kinds of work were being done, opened on to the roof, so that at times the air over the roof probably contained large amounts of nitrous acid.

In the course of my inquiry samples of flour milled by firms who possess no bleaching plants have been found to contain as much as 0·5 part per million of nitrites (expressed as $NaNO_2$). On the other hand samples known to have passed through a bleaching apparatus have been obtained which gave amounts of nitrites lower than 0·5 part per million.

For practical purposes, however, it may be presumed with some probability that when more than one part of nitrites per million is present the flour has been bleached; with quantities much above this the inference of bleaching amounts to practical certainty. On the other hand, when flour has been submitted to the Griess-Ilosvay test and has been found free from nitrites, there can be little doubt as to its being unbleached.

Nitrites in Flour Sold in this Country.—As a result of examinations of samples of flour taken under the Sale of Food and Drugs Acts, 12 samples of flour purchased in shops in Glasgow were recently reported by the Public Analyst for the County of Lanark to contain nitrites in varying amounts estimated at from 0·01 to 1·2 parts per million.

The Public Analyst for the County of Nottingham recently examined a number of samples of flour and found them to contain an average of six parts per million of nitrogen peroxide. The Public Analyst for the County of Chester examined, in 1909, 22 samples of flour of which 14 were bleached. The Public Analyst for Kingston-on-Hull, as a result of the examination of 20 samples of flour, found that 11 contained nitrites.

Information has kindly been supplied to me by medical officers of health and several other public analysts of the result of examination for nitrites, by the Griess-Ilosvay test, of flours purchased at retail shops or sold to bakers in different parts of England. Out of 157 samples 40 were reported to be free from nitrites and 117 contained nitrites. Of these 117 samples—

72	contained	more than	0·5	parts of $NaNO_2$	per million	of flour.
35	"	"	1·0	"	"	"
4	"	"	2·0	"	"	"
2	"	"	3·0	"	"	"

None contained more than 4 parts per million.

Samples of flour were also taken by me at different mills immediately after being bleached and their nitrite content estimated. Out of 13 such samples only one contained as much as 4 parts per million of nitrites (expressed as $NaNO_2$), one contained 3·5 parts per million, one contained 2 parts per million, four contained 1·5 per million, and the remaining six samples 1 part, or less, of nitrites per million. The amount of nitrite present in these samples is approximately of the same order of magnitude as that found in the nitrite-containing samples obtained in the ordinary way in retail shops and of bakers throughout the country.

These results would appear to show that, at the present time, flour bleached in this country contains somewhat less nitrite than is given in recorded results of analyses of flour bleached in the United States and Canada.

Alway and Gortner(1) examined a number of samples of bleached flour in the United States and found the average amount of nitrites present in the samples to be 6·3 parts per million. Hale(7) also asserts that in the United States the average amount of nitrite-nitrogen in bleached flour is about one part per million, *i.e.*, about 5 parts of nitrite (as sodium nitrite) per million of flour, and Wesener and Teller(19) consider this to be the average nitrite content of bleached flour. In Bulletin 206 of the Laboratory of the Inland Revenue Department, Canada, a series of analyses of flour is given in which amounts of nitrites as high as nine parts per million have been detected. Several samples contained amounts above four parts per million. Out of 223 samples, however, 148 gave no reaction for nitrites.

Nitrites in Imported Flour.—A number of consignments of flour have recently been sampled on arrival in this country from abroad (chiefly the United States and Canada), and have been examined by Dr Monier-Williams for the presence of nitrites. Out of 70 such samples 13 were found to be free from nitrites and 57 contained nitrites. Of these 57 samples—

34	contained	more than	0·5	parts of $NaNO_2$	per million	of flour.
24	„	„	1·0	„	„	„
11	„	„	2·0	„	„	„
4	„	„	3·0	„	„	„
2	„	„	4·0	„	„	„

None contained more than 4·5 parts per million. The amount of nitrite present in these samples is approximately of the same order of magnitude as that found in samples bleached in this country.

5.—The Addition of Other Substances to Flour. Flour "Improvers," so-called.

As my inquiry progressed it became clear that, besides the question of bleaching, other additions to flour had to be considered, in particular the following:—

Water.—In some cases water may be added to flour in the form of a very fine spray by means of an "atomiser," or other suitable apparatus. The spraying may take place upon the finished flour or at various stages during the milling process; usually the latter, since more intimate incorporation is thereby ensured. This treatment is stated to improve the baking qualities of wheat in certain cases and to facilitate the milling processes. Incidentally it is possible in this way to add about one or two per cent. of moisture to wheat already containing 15 per cent. of water. This represents a considerable gain to the miller. I am informed that in some cases glycerine may be added to the water to assist the flour to retain the added moisture.

Phosphates and other "Improvers."—The work of Wood and others(21)(22) indicates that the properties of gluten can be modified by the addition of certain salts and acids to flour. Wood also states that the ash of strong wheats is richer in phosphates than that of weak wheats. Endeavours are being made at the present time to improve the baking qualities of certain flours by treating them with various salts and acids with the object of improving the quality of the gluten so as to increase the strength and water-absorbing capacity of the flour. One of the results of this is that the number of loaves which it is possible to obtain from a sack of flour is increased. By this means it is hoped that the value, from the trade point of view, of weak wheats, such as English wheat, may be increased. Many of these substances, in addition to being "improvers" in the above sense, stimulate the activity of yeast in bread-making.

A process is in use whereby solutions of acid potassium phosphate, diastase and other substances may be sprayed on the flour during or after milling by means of an apparatus similar to that already mentioned for spraying flour with water. The process is considered specially suitable for country millers who have ready access to English wheat, as it is said to enable them to mill a mixture containing a large proportion of English wheat and a correspondingly small amount of strong foreign wheat. It is said, however, that considerable skill and judgment is required in order to obtain good results, and for this amongst other reasons the process has hardly if at all been adopted by English millers. Flour from durum wheat is stated not to be improved.

A certain chemical manufacturer proposes to supply millers and bakers with a mixture of acid potassium phosphate and wheat diastase to be added to flour in the proportion of one or two pounds of the mixture to each sack (280 lbs.) of flour. It is stated that experiments with the above mixture have shown that the flour from certain wheats can be greatly improved as regards strength and water-absorbing capacity.

Another "improver" has lately been introduced. It consists of acid potassium and magnesium phosphates mixed with flour; the mixture contains phosphates and flour in about equal proportions. The potassium and magnesium phosphates are mixed in the proportion in which they occur in the ash of wheat. The manufacturer recommends that 1 to 1½ lbs. of the "improver" should be added to each sack of flour (280 lbs.). He considers that this preparation is most effective as an improver. As an inducement to use this preparation, it is represented to the miller that he can use a larger proportion of cheap wheats, and to the baker that as many as 10 to 14 extra 2 lb. loaves can be obtained from a sack of flour by its use.

Manufacturers of acid calcium phosphate have largely advertised this substance to millers and bakers as a desirable flour improver, by means of which the baking qualities and also the colour of the flour may be improved. One such firm issues instructions to add the "improver" at the rate of 2 lbs. per sack of flour (280 lbs.). They guarantee that the flour to which their improver has been added "will be passed as pure wheaten flour by any and every analyst to whom it is submitted for analysis, and that it can be sold as such[1]." They state also that it improves the strength of the flour to the extent of at least 1*s.* 6*d.* per sack and the colour by 1*s.* per sack in value.

Various grades of "acid phosphate" are manufactured—the higher grades by the treatment of bone ash with phosphoric acid, the lower grades by acting on bone ash with sulphuric acid. In the latter case the "improver" may contain 50 per cent. or more of calcium sulphate and an appreciable amount of arsenic. A sample of "improver," recently examined by the public analyst for Kingston-on-Hull, was found to contain 0·07 grain of arsenic per pound, which is seven times the maximum limit suggested by the Royal Commission on Arsenical Poisoning[2]. Both high and low grades of acid calcium phosphate are being offered as flour improvers.

Many millers have experimented with acid calcium phosphate, but it is difficult to ascertain how many of them actually make a practice of adding it to their flour. It is stated that the number is large, and that in many cases the acid phosphate is secretly introduced into the mills for the purpose, or is added on separate premises. In one mill which I visited acid calcium phosphate was being added to, and intimately mixed with, the whole flour output (30 sacks per hour) by means of a specially contrived mixing machine in the proportion of

[1] A method of detecting added acid calcium phosphate in flour and bread has recently been described by G. Curtel, *Annales des Falsifications*, No. 21, 1910, p. 302.

[2] See Final Report of the Royal Commission on Arsenical Poisoning, page 50, 1903 [Cd. 1848].

rather more than 1¼ lbs. per sack of flour (280 lbs.), so that the finished flour contained about 0·45 per cent. of acid calcium phosphate. Other millers whom I visited made a practice of adding acid calcium phosphate to their flour in much the same way.

Phosphoric Acid.—The addition of acid in a suitable manner to flour is also said to improve its baking qualities, loaves made from such flour being larger and whiter than those made from untreated flour. The acid preferred is phosphoric acid, and it is claimed that if the process is carefully carried out a great improvement in the baking qualities can be effected in flour made from weak wheat.

In a mill where this treatment is being commercially applied phosphoric acid is allowed to drip slowly into a jet of air and steam; the resulting atomised mixture enters an agitator, where it meets a continuous stream of flour with which it becomes thoroughly incorporated. The introduction of about 0·1 per cent. of phosphoric acid is aimed at, and the difficulty of detecting this by chemical analysis renders the process additionally attractive.

A firm engaged in the chemical industry is interested in an "improver" consisting of semolina which has been treated with phosphoric acid. Thirty parts of phosphoric acid (sp. gr. 1·450) are mixed with 100 parts of semolina, which is then dried and ground. This preparation is directed to be used in the proportion of about 1½ lbs. to a sack of flour. The firm promise a great increase in the strength and water-absorbing capacity of the flour as a result of this treatment, which, they say, will result in a saving to the miller of 1*s.* to 1*s.* 6*d.* on each sack of flour.

Other Substances.—Ozone has been tried for bleaching purposes, but according to Fleurent(6) it possesses no bleaching properties, and imparts a disagreeable flavour to the flour. Brahm(5) has confirmed this, and has found that the baking qualities of flour are adversely affected by treatment with ozone. Molinari and Soncini(12) have shown that ozone attacks and destroys olein, a constituent of the oil of flour.

Sulphur dioxide has been proposed as a bleaching agent for flour, and according to Avery(3) and Alway and Gortner(1) is effective as far as mere bleaching is concerned. It cannot, however, be used for this purpose, as it destroys the cohesiveness of the gluten.

Flour has also been treated with sulphuryl chloride, sulphur trioxide and chlorine, and other similar mixtures, but I am informed that the results have not been encouraging. Sulphuryl chloride is stated to improve the strength of flour, but the sulphur trioxide and chlorine mixture is uncertain in action, and is usually without any beneficial effect on the baking qualities of flour. In practice the odour which these substances impart to the flour precludes their use as "improvers."

In some recent patents it is proposed to treat flour with phosphorus trichloride, pentachloride or other halogen compounds of phosphorus, or with a mixture of these and sulphur trioxide, nitric acid, nitrous acid, iodic or other halogen acids; also formic, acetic, propionic or benzoic acids, alcohol, aldehydes, or ketones, with the object of strengthening the flour and improving its baking qualities. It has further been proposed to treat flour with phosphorus pentoxide, phosphorus bisulphide, and phosphorus pentasulphide, and the process has been patented.

Although much experimentation of an empirical kind is proceeding, in the course of which a variety of heterogeneous substances may be added to flour, it may be said that apart from nitrogen peroxide the only substances whose use as yet has been attended with any measure of commercial success are certain acids and salts, more particularly phosphoric acid and phosphates.

6.—Bleaching and so-called Flour "Improving" in relation to Public Health and the General Interests of the Consumer.

Bleaching by Nitrogen Peroxide.

As in the case of preservatives and other deleterious substances added to food in small quantities, great difficulties beset the experimental investigation of the effect of small amounts of nitrogen peroxide, such as are used in bleaching, on the properties of flour and on the health of the consumer.

Although a number of investigations have been undertaken on this subject, the results obtained have been in many respects contradictory and inconclusive. Gluten and bread, prepared from bleached and unbleached flour respectively, have been compared as regards their digestibility by pepsin and trypsin *in vitro* with divergent results. Ladd and Bassett (9) have found that gluten from bleached flour is more slowly digested by these enzymes than is gluten from normal unbleached flour, and a similar but less marked difference has been observed in the case of bread made from bleached and unbleached flour.

Gustav Mann (16) states that gluten from unbleached flour is digested by pepsin about twice as rapidly as gluten from bleached flour containing 7 parts per million of nitrite-nitrogen. Hale(7) has also found that bleaching delays the peptic digestion of flour. Snyder(15), however, found no difference between the behaviour of bleached and unbleached flour towards pepsin.

Halliburton(8) has found that traces of nitrites exert a distinctly inhibiting effect on both proteolytic and amylolytic enzymes. One part of sodium nitrite in 8000 was found to inhibit peptic digestion entirely, and one part in 32,000 to decrease the activity of the enzyme to one-seventh of its normal activity. Similar results were obtained in the case of the salivary digestion of starch. From further experiments Halliburton concluded that treatment of a protein with even minute traces of nitrous acid alters it in such a way as to render it less readily susceptible to the action of digestive enzymes. Hale(7) has found that sodium nitrite in a dilution of one part in 5000 to 200,000 has a distinct inhibitory action on gastric digestion.

Feeding experiments have also been performed to ascertain whether bleached flour can exert any observable effect upon the living animal. Ladd and White(11) administered watery and alcoholic extracts of bleached flour to rabbits by the mouth, and death rapidly supervened. This occurred even though the extracts were neutralised by sodium carbonate before administration. On post-mortem examination the viscera were congested, and the condition of the stomach was such as would result from the administration of an irritant poison. It was suggested that a toxic substance—possibly a diazo-compound—produced in the flour as a result of bleaching was responsible for the death of the rabbits and for the post-mortem appearances. Other observers(19) (20) have repeated these experiments, and have failed to obtain the results above described. Wesener and Teller(19) have also fed a number of rats for some months upon bread and biscuits made from bleached flour without apparently affecting their health in any way. Hale(7) found that alcoholic extracts of overbleached flour were devoid of any marked toxic action when introduced into the stomach.

In this connection, it should be noted that of all biological methods of investigation those involving artificial digestion and feeding experiments require the observance of very special precautions if reliable results are to be obtained. Disregard of such precautions may account for many of the discrepancies which appear in the work done on this subject. Even when due care is exercised, the results obtained from such experiments may be, and frequently are, inconclusive. Numerous disturbing factors inherent in experiments of this kind may obscure or lead to erroneous interpretation of results which, from the nature of the investigation, often cannot be expected to be marked.

Ladd and White's results, if confirmed, would indicate the existence of a serious danger of acute poisoning from bleached flour. This point was investigated by Dr A. Harden at the Lister Institute. Dr Harden shows that under the conditions in which his experiments were conducted no obvious effects were produced on animals fed with highly bleached flour or with aqueous extracts of such flour.

He also shows that bleaching has a distinctly inhibitory effect on peptic digestion but no observable effect on the pancreatic (proteolytic) digestion of flour. Dr Monier-Williams has demonstrated that sodium nitrite exerts no inhibitory effect on the salivary digestion of starch, but in the case of starch treated with nitrogen peroxide, digestion was greatly retarded. Bleaching was also found to exercise an inhibitory effect on the salivary digestion of flour.

As regards the action of nitrites on the system, it should be remembered that nitrites, when administered as drugs, produce various effects, amongst which disturbance of the heart and vascular system is prominent. The amounts of nitrite introduced by bleached flour would be of a much lower order than those taken when nitrites are given medicinally[1]. Statements have been made, however, by medical practitioners that an appreciable effect may be produced by quite small doses of nitrite: Gustav Mann(16) points out that quantities such as half a grain (32 milligrammes) of nitrous acid may be harmful to some individuals. What may be the physiological or pathological effect of ingestion of even smaller doses when taken day by day throughout many months or years it is impossible to say; there is no evidence on the matter, and it would be very difficult to obtain any. It cannot, however, be regarded as desirable that minute doses should be ingested day by day of a drug which in larger single doses has a marked action on the vascular system.

It would appear from experimental and other considerations to which reference has already been made, that, apart from the addition of nitrites, the constitution of flour may be altered by bleaching. Dr Monier-Williams has clearly shown in his report that the oil of flour undergoes a marked change: it has been suggested by Folin(16) that nitration of the flour oil, if it occurs, might entail risk to health, since absorption of the oil and its oxidation in the tissue might occur,

[1] The pharmacopœial dose of sodium nitrite is 1 to 2 grains, and of nitroglycerine (in Liquor Trinitrini) $\frac{1}{200}$ to $\frac{1}{50}$ grain.

resulting in the liberation of nitrites. Ozone produced together with nitrogen peroxide in certain bleaching processes also exerts a markedly destructive action on olein, one of the constituents of flour oil. In spite of assertions to the contrary, there seems to be evidence pointing to the possibility of the protein constituents of flour being adversely affected as the result of bleaching. Dr Monier-Williams has shown that the solubility of the proteins and also of the carbohydrates is increased by such treatment. These changes in the oil, the protein and the carbohydrates are, of course, more marked when flour is overbleached, and though overbleaching is not likely nowadays to occur throughout the whole of the flour, local overbleaching may take place, portions of flour adhering to the sides of the agitator and flour spouts may become overbleached and contaminate to some extent, at any rate, the rest of the flour.

Looking to the above considerations, it may be concluded that the alterations in, and the additions to, flour which result from a high degree of bleaching by nitrogen peroxide cannot be regarded as free from risk to the consumer, especially when regard is had to the inhibitory effect of the bleaching agent on digestive processes and enzymes. Even in the case of flour which is bleached to the small extent which is at present ordinarily practised, it would in present knowledge be unwise to conclude that the process is attended by absolute freedom from risk. The fact that bleached flour has been shown to be something more than natural flour, the colour of which has been modified, is also of importance in considering whether bleached flour may properly be represented as genuine flour.

The practice of bleaching being open to these objections, it remains to inquire whether the consumer, who at present is seldom aware that his flour has been bleached, or that his bread is made from bleached flour, can be said to obtain any compensating benefit. To this a negative answer must be given.

Apart from any dietetic considerations a large number of people desire bread of exceptional whiteness, and it is reasonable to suppose that what is demanded by those who prefer such bread is an article made from flour, the whiteness of which is due to its being prepared from specially selected wheats by the elaborate mechanical separation and "purification" of modern milling methods. Few people would carry their approval of whiteness to the extent of requiring naturally dark flour to be chemically treated.

It should be noted in this connection that in some countries where special attention has been given to the bleaching of flour steps have already been taken to discourage or prohibit the practice. I have already referred to the decisions on the subject by the Board of Food and Drug Inspection of the United States Department of Agriculture. Under the laws against adulteration in certain of the Australian States, the official definitions of what may properly be described as flour exclude flour which has been treated by bleaching, and the official definition of flour recommended by the Departmental Conference on uniform standards for foods and drugs in the Commonwealth and States of Australia (1910) to be applied throughout the Commonwealth, contains a provision expressly excluding the bleaching of flour. In Switzerland the bleaching of flour has been prohibited by Article 61 of the Loi fédérale sur le commerce des denrées alimentaires et de divers objets usuels (Du 8 décembre, 1905).

So-called "Flour Improvers."

The use of these substances is discouraged or prohibited by the official requirements of the countries above referred to, and these articles can hardly be regarded as proper constituents of what is represented to be genuine flour in this country.

Those interested in these preparations advocate their use on the grounds that they add nothing to the flour that is not normally present therein, and that they increase the lightness and improve the quality and appearance of the loaf, and also permit of more loaves being made from a given quantity of flour. The first of these contentions is based on the assumption that phosphorus in flour is present in the form of phosphate, chiefly potassium phosphate. This is not so; it is true only of the ash of flour. A large portion of the phosphorus in flour is present in organic combination, and experimental evidence exists which would seem to indicate that phosphorus in this form may possess a dietetic value quite different from inorganically combined phosphorus. This is recognised by certain millers, and it is suggested that such organic phosphorus compounds may be formed when solutions containing phosphates are intimately mixed with flour in the form of a fine spray from an "atomiser." No evidence is adduced in favour of this contention, and it may suffice to say that the formation of complex organic phosphorus compounds in this way is contrary to experience.

The second advantage claimed, namely, the increased loaf production from a given quantity of flour is one which will appeal only to the miller and baker. The gain in production is due to the increased amount of water which the flour absorbs and to the increase in volume of the dough, resulting from the improved elasticity of the gluten. This naturally means a diminution in the actual amount of flour in each loaf, and, consequently, in nutritive value, so that the consumer in this respect loses by the treatment.

The protein content of flour is an important matter from the standpoint of nutrition, especially where bread enters largely into a diet. Flour from weak wheats, which are generally poor in gluten, contains less protein than flour from strong wheats which are rich in gluten. But by the use of "improvers" flour from weak wheat is made to simulate flour from a stronger wheat, although as regards protein content it is inferior to the flour which it imitates.

With regard to other substances which have been represented as "improvers," it may be said that the indiscriminate addition of powerful chemical substances such as hydrofluoric acid, phosphorus pentachloride, and the oxides and sulphides of phosphorus to flour is most dangerous.

At the present time many millers are unaware of the nature and composition of the "improvers" which they add to flour, and are content with the assurance of chemical manufacturers that the preparations offered to them constitute, in all respects, desirable additions to flour. If the use of "improvers" became general, there can be little doubt that manufacturers of chemical products would persuade millers to treat their flour with preparations often of a questionable nature, sold under attractive and, possibly, misleading names. There can be no doubt also that competition amongst chemical manufacturers would eventually result in millers being supplied with low grade articles liable to contain dangerous impurities.

It must also be remembered that the addition of foreign substances may not be limited to the miller, but that each person through whose hands flour passes (*e.g.*, the flour factor and the baker), may add to it a different substance or more of what the miller has already added. In this way flour, when it reaches the consumer, may have departed considerably from its original state of purity.

The increasing activity which is now being displayed in the use of different articles as additions to flour must be regarded with considerable apprehension. It does not appear desirable that such an indispensable foodstuff as flour, the purity and wholesomeness of which are of first importance to the community, should be manipulated and treated with foreign substances, the utility of which, from the point of view of the consumer, is more than questionable.

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NOTE:—Numerous papers on wheat and flour were also read at the Seventh International Congress of Applied Chemistry, London, 1909, and at the British Association for the Advancement of Science, Winnipeg, 1909.