

STUDIES ON STARCHES FROM INDIGENOUS GRAINS AND TUBERS.

PART II. JOWAR STARCH.

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Jowar (*Sorghum vulgare*) is grown in various parts of India, especially in Bombay and Madras Presidencies, and covers an area of over twenty million acres. The grain is consumed by certain classes of people, but it serves mainly as cattle food. The starch content of the grain varies from 55–64 per cent. depending on variety. The starch is of good quality and is quite suitable for use in the textile industry. In view of this and the comparative cheapness of the grain, a systematic enquiry on the preparation of starch from jowar was undertaken.

There are two possible methods of preparing starch from a grain of the type of jowar. One is to use the grain and the other, to use only the endosperm. Archbold (*Jour. Soc. Chem. Ind.*, 1902, 21, 4) developed a method for the manufacture of corn starch using the whole grain but subsequently it has been found advantageous (Williams, *Amer. Chem. Abst.*, 1921, 15, 3224) to first remove the embryo which makes a valuable by-product. The germ oil finds application in the manufacture of soap and other industries, while the residual cake makes an excellent feeding stuff. The related processes are however tedious and involve the use of expensive machinery. Moreover, corn contains about 4 per cent. of fat, while jowar contains only about 2 per cent. In view of this and the need for developing an inexpensive method that could be adopted even on a small scale, the germ was not separated in the present enquiry.

EXPERIMENTAL.

The grains were obtained from the local market and, after drying, were powdered to pass the 90-mesh sieve. The following is the proximate composition of the powder as determined by the A.O.A.C. (1930) methods:—Moisture, 12.0; Crude protein ($N \times 6.25$), 8.2; Sugars (as glucose), 4.5; Starch, 56.0; Fat, 1.6; and Ash, 1.5 per cents. respectively. The powder was then treated with different concentrations of caustic soda, four parts of alkali being added to one part of powder in each case. The suspensions were then allowed to stand for

48 hours with occasional stirring. The purity of the samples thus obtained was then determined (*A.O.A.C.*, 1930), after making them alkali-free by repeated washing on the centrifuge and finally drying in a current of hot air (about 45°). The results are given below (Table I). As the quality of the product was poor, the yields have not been given.

TABLE I.

Alkali per cent.	0.6	0.8	1.0	1.2
Percentage of starch in the preparation	88.0	88.4	90.2	89.2

The results show that the samples were not sufficiently pure for commercial purposes. Moreover, it was observed that at the higher concentrations of alkali (1.0 and 1.2 per cents.) there was partial gelatinization of starch. The low purity was traced to the fact that there was no proper penetration of alkali in any of the cases.

With a view to eliminating this difficulty, the powder was allowed to undergo fermentation prior to alkali treatment. The procedure consisted in mixing the powder well with ten parts of water and allowing the suspension to stand for four days. The scum that formed at the top was removed at intervals. The suspension turned steadily acid, finally attaining a pH value of about 5.0. It was then centrifuged, and washed free from acid and dried. The preparation thus obtained was treated with different concentrations of alkali and allowed to stand for 24 hours. After washing free from alkali, the samples were dried and their purity determined as before (Table II).

TABLE II.

Alkali per cent.	0.4	0.5	0.6	0.7	0.8	0.9
Percentage of starch in the preparation	90.6	91.3	92.0	92.6	94.5	94.8

Although a fairly pure product can be obtained by the above method, it was yet found that dry sieving of powdered jowar was tedious and time consuming. Moreover, the powder frequently choked up the pores of the sieve. The product was of light yellow colour which could not be improved by bleaching.

Wet grinding of the grains was then adopted. The magma thus obtained was then washed on a 100-mesh sieve and the starch milk thus obtained, centrifuged. Two distinct layers can be separated in this manner. The top layer consisted of glutinous starch associated with fat and some cellulosic material, but the bottom one consisted mostly of pure starch. The purity of the crude preparation thus obtained was 89.6 per cent. With a view to purifying it, it was subjected to treatment with alkali of various concentrations using four parts of alkali for every part of dry powder. After 24 hours, the samples were washed free from alkali and their purity determined after drying as before (Table III).

TABLE III.

Alkali per cent.	0.5	0.6	0.7	0.8	0.9
Percentage of starch in the preparation	95.8	96.0	96.8	97.0	96.7

With a view to determining the minimum quantity of alkali required for purifying the starch, the following experiment was carried out. The starch powder (89.6 per cent.) was treated with increasing proportions of 0.7 per cent. alkali which was found to be most suitable from the previous set of trials. After being steeped for 24 hours, the samples were washed free from alkali, dried and their purity determined (Table IV).

TABLE IV.

Proportion of alkali to powder	3 : 1	4 : 1	5 : 1	6 : 1	7 : 1
Percentage of starch in the preparation	93.8	96.2	96.7	97.7	97.8

It may be seen from the above that treatment with 4 parts of alkali is quite sufficient for obtaining a fairly pure preparation suitable for most textile operations.

It was found, however, that wet grinding of fresh grains was slow and tedious. The preliminary centrifuging was also time consuming. It was considered necessary, therefore, to overcome the defects and to quicken the operations.

To facilitate the grinding, the grains must first be softened. They were steeped, therefore, in an equal volume of dilute sulphurous acid

(about 1 per cent. of SO_2) for 3 to 4 days. It was expected that the antiseptic would prevent the growth of mildew and discourage fermentation. The grains were then washed till acid-free and ground to make a thick suspension with water. The suspension was then screened through 90-mesh sieve. The residue left on the sieve was mostly skin. The suspension was allowed to stand overnight. The following morning, the clear supernatant was syphoned off. The solids were found to have settled in two distinct layers which were easily separated. The top layer which was associated with the major part of the fat and protein was brownish in colour. The bottom one was mostly starch and yielded, on drying, a product which was 92.4 per cent. pure.

Further purification.—The dry powder obtained from the bottom layer was treated with varying concentrations (0.2–0.6 per cent.) of caustic alkali (4 parts of alkali to 1 part of starch). The alkali suspensions were kept stirred for 6–8 hours for, otherwise, the starch tended to quickly settle down and form a sticky, compact mass. The suspensions were then allowed to stand for 6 hours, preferably overnight, after which the coloured supernatant was syphoned off. The precipitated starch was then passed through a 120-mesh sieve, washed free from alkali (by repeated washing, settling of the starch and draining off the supernatant liquid) and finally dried. The purity of the products thus obtained was determined in the usual way (Table V).

TABLE V.

Alkali per cent.	0.2	0.3	0.4	0.5	0.6
Percentage of starch in the preparation	93.8	95.2	95.4	95.3	95.7

It may be noted that the use of 0.3 per cent. alkali yielded nearly as good results as any of the higher concentrations that were tried.

Treatment of the top layer.—The slimy mass which constituted the top layer was stirred with water and centrifuged at 2,000 R.P.M. in a cloth-lined machine. It was found that the materials had separated into two layers. The one at the top yielded, on further drying, a dark brown cake corresponding to 2.5–3.0 per cent. on the weight of the original grain. The cake contained 36.0 per cent. of ether extractives and 37.6 per cent. of crude protein. The oil present in the extract is semi-drying one and is of golden yellow colour. It closely approaches maize oil in its properties and can therefore be used either as an edible oil or in the manufacture of special kinds of paints and varnishes (*Bull. No. 7, 1933, Department of Industries, Bombay Presidency*).

The other layer which settled at the bottom was treated with alkali for the separation of starch. Alkali solutions of different concentrations (0.6–0.9 per cent.) were used, maintaining the same proportion of alkali to crude starch as before. The suspensions were allowed to stand for 24 hours stirring at intervals and allowing to settle. The highly coloured supernatant liquid was syphoned off and washed free from alkali. It was then passed through a 120-mesh sieve to remove suspended impurities, then centrifuged and dried. The purity of the samples was then determined as before (Table VI).

TABLE VI.

Alkali per cent.	0.6	0.7	0.8	0.9
Percentage of starch in the preparation	91.2	91.8	92.6	93.0

The preparations were slightly coloured. The low purity of the samples was traced to the presence of cellulosic material and some unopened cells which had separated along with the starch.

Among the concentrations of alkali that were tried, 0.8 per cent. was the most satisfactory. Separation of starch from associated impurities was carried by lixiviation.

Purification by lixiviation.—This was done by adding 5 to 6 times the volume of water and then stirring up the whole suspension. After being allowed to stand for 3 to 4 minutes, the milky supernatant containing the bulk of starch was quickly syphoned off. By repeating the process 5 to 6 times, all the starch was lixiviated out. The different collections were mixed together and allowed to settle overnight. The supernatant liquid was then syphoned off and the starch settling at the bottom dried in the usual way. The purity of the preparation thus obtained was found to be 95.5 per cent.

Outline of procedure for the preparation of jowar starch.—The grain is first passed through a 16-mesh sieve to remove small stones and dust. It is then washed under the tap on the sieve itself and steeped in sulphurous acid (equivalent to approximately 1 per cent. SO_2) and left for 3 to 4 days till it becomes soft enough for grinding. The swollen grains are then washed to remove adhering acid, ground in an edge-runner to form a homogeneous pulp and washed on a 90-mesh sieve. The suspension is then allowed to settle overnight. The following morning, the supernatant is syphoned off and the two layers separated. The bottom layer (A) is almost white, while the top one (B) is brownish in colour.

The layer (A) is directly treated with alkali solution. The strength of alkali is so adjusted that the final concentration may correspond to 0.3 per cent. The suspension is kept well stirred for about 8 hours and then allowed to settle. The supernatant liquid is syphoned off, then mixed with fresh quantity of water and passed through a 120-mesh sieve. The starch is washed and allowed to settle. The operation is repeated two or three times till the suspension becomes free from alkali. It is dried first by rotation in a hydro-extractor and then in a current of air (temp. about 45°). The yield of starch obtained in this way corresponds to about 42-48 per cent. (on air dry basis), calculating on the total weight of the grains used.

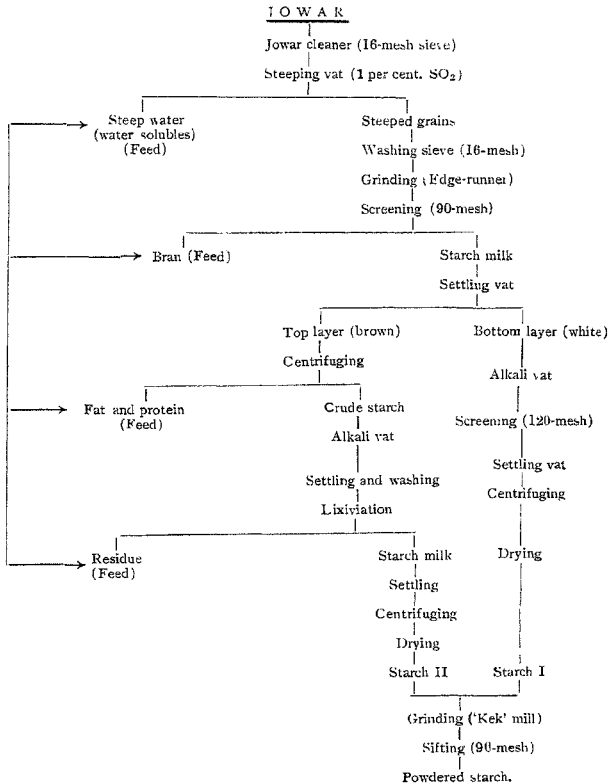
The layer (B), when centrifuged, again separates into two layers. The top layer (C) being mostly protein and fat, is utilised as fodder, while the bottom one (D) is treated with an alkali solution in such a way, that the final concentration would correspond to 0.8 per cent. The suspension is left for 24 hours with stirring at intervals. The supernatant liquid is syphoned off after the starch has settled down. By repeated washing and settling, this is made free from alkali and the starch lixiviated out. The milky suspension thus obtained is allowed to settle overnight. The clear suspension is then syphoned off and the starch dried first in the centrifuge and then in a current of air as before. Thus a further yield corresponding to about 10 per cent. on the dry weight of the jowar is obtained.

Starting with 100 lbs. of jowar, one can obtain about 57 lbs. of starch and about 20 lbs. of feed. The starch is perfectly white and requires no bleaching.

About 3 cwts. of starch thus prepared was sent to a leading firm of textile manufacturers in South India for trial. They have reported it to be a very good-sizing starch.

Some preliminary trials with other varieties of jowar.—These experiments were carried out with three varieties of jowar, one of which was obtained locally, while the other two were got from the Ceded Districts, Madras. Of the latter, one was coloured red. The starch obtained from it was also coloured and necessitated the use of strong bleaching agents to obtain a white product. The other variety from Ceded Districts yielded a white but rather coarse product which was not so satisfactory as that obtained locally. All the three varieties were rather poor in starch, the best containing only 56 per cent. If the grain is to compete successfully with corn as a raw material, it would be necessary to use varieties containing at least 64 per cent. of starch. A number of other indigenous varieties are therefore being examined with a view to selecting one suitable for the purpose.

MANUFACTURE OF JOWAR STARCH.

(Process Flow Sheet)

SUMMARY.

1. Treatment of dry, powdered jowar with caustic alkali (0.6–1.2 per cent.) followed by centrifuging yields a coloured product containing under 90 per cent. of starch.

2. Preliminary fermentation accompanied by frequent removal of scum helps to remove a large part of the fat, proteins and colouring materials. Subsequent treatment with alkali (0.8 per cent.) followed by centrifuging yields a product of nearly 95 per cent. purity.

3. Steeping the grain in sulphurous acid (1 per cent.) followed by wet grinding yields a paste which can be more easily handled than dry powder. On centrifuging the paste, the major part of the starch separates out as the bottom layer. On further treatment with alkali (0.3 per cent.) and recentrifuging, a product of nearly 97 per cent. purity is obtained. The top layer is more difficult to handle, but yields starch of nearly the same purity on treatment with 0.8 per cent. alkali followed by centrifuging.

4. The details of procedure for preparing jowar starch are given. A scheme of operations for production on a factory scale is also outlined.