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(54) **Process for steeping corn and steepwater having a low reducing sugar content made therefrom**

Verfahren zum Quellen von Mais und daraus hergestelltes Quellwasser mit einem niedrigen Gehalt an reduzierendem Zucker

Procédé de trempage du mais, liquide de trempage ainsi obtenu contenant une faible teneur en sucres réducteurs

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(56) References cited:
JP-A- 2 211 860

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EP 1 044 988 B9

Description

BACKGROUND OF THE INVENTION

Field of the Invention

[0001] The present invention relates to an improvement in the wet milling process for separating and recovering products from corn. More particularly, the present invention relates to an improved steeping process which results in improved millability giving higher yields of starch and in a steepwater having a low reducing sugar content.

Description of the Related Art

[0002] In the usual processing of corn, the whole corn is steeped in water containing sulfur dioxide at an elevated temperature and acid pH for an extended period of time. During the course of the soaking, the soluble substances are extracted from the corn grain to the soaking water where they ferment due to the presence of lactic acid bacteria in the water. Variables such as soaking time, SO_2 levels, water removal rates, temperatures, etc. are used to promote separation. The resulting steepwater is used as a fermentation medium to produce a wide variety of products such as enzymes, pharmaceuticals, etc. Due to the many variations in the process, the quality of corn steepwater found in the market varies widely.

[0003] An early description of a simple steeping process is described in U.S. Patent RE 18,775 where acidulated water is added to a single steep tank. Following separation of the starch, the water is re-used in the process. Other early descriptions of steeping processes involving the recycling of the steepwater back to the process are found in U. S. Patents 58,824; 1,655,395; and 1,960,985.

[0004] U.S. Patent 2,232,555 describes a process in which corn is soaked in acidified water having a pH of 4.5 to 6.9 at a temperature of 51.7°C (125°F) to 62.8°C (145°F) for about 5 to 15 hours to provide an extract containing substantial quantities of water soluble carbohydrates or sugars.

[0005] U.S. Patent 4,359,528 describes a process for producing steepwater having a high amino acid content and useful in the production of penicillin. The process utilizes decreasing temperatures over a series of silos and high cycling ratios of water to corn. A pH of 3.95 to 4.2 is reported.

[0006] U.S. Patent 4,980,282 describes a process in which the steepwater is separated from the process, incubated to develop a biomass and the developed biomass treated at a temperature between 40°C to about 48°C at a pH of at least about 3.5.

[0007] While producing steepwater of varying degrees of usefulness, a process has now been found that provides a steepwater having the low reducing sugar content necessary for use as a fermentation medium while providing high yields of starch.

SUMMARY OF THE INVENTION

[0008] According to this invention there is provided a process for producing corn steepwater comprising

(a) introducing fresh steepwater containing from 1000 to 2200 parts per million sulfur dioxide to corn;
(b) steeping the corn for a steeping time greater than 10 hours at a temperature from 47.8 °C (118°F) to 51.7 °C (125°F),
(c) separating the steepwater from the corn at a rate of 7.6-15.1 Litres (2 to 4 gallons) per 35.2 litre (bushel) of corn;
(d) adjusting the pH to from 4.2 to 5.5 at a time between 4 hours after the fresh steepwater is introduced to 1 hour before the separation of the steepwater from the corn ;
(e) maintaining the separated steepwater at a temperature from 47.8°C (118°F) to 51.7 °C (125°F) for between 10 to 40 hours; and
(f) evaporating the corn steepwater.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] For a fuller understanding of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawings, in which:

FIG. 1 is a diagrammatic side elevation view of a battery of silos used in a corn steeping process;
FIG. 2 is a chart showing starch yield as a function of steep time and time of pH adjustment;
FIG. 3 is a chart of curves showing incubation at different temperatures;

FIGS. 4 and 5 are graphs showing percent reducing sugars in steepwater in the incubation tanks before and after pH adjustments in the steeps.

DETAILED DESCRIPTION OF THE INVENTION

[0010] In carrying out the process, corn grains, placed in silos, are contacted with steepwater containing from 1000 to 2200 parts per million sulfur dioxide to corn. The corn is steeped for greater than 10 hours and preferably between 20 to 48 hours at a temperature from 47.8°C (118° F) to 51.7°C (125°F). Steeping can be carried out in a batch process or in series as shown in Fig. 1. When carried out in series, fresh steepwater is introduced successively into each of the silos in the series.

[0011] In a normal steeping process, the pH is 3.8 to 4.1. It has been found in the process of this invention that adjusting the pH to from 4.2 to 5.5 at a time between 4 hours after the fresh steepwater is introduced initially to 1 hour before the separation of the steepwater from the corn followed by maintaining the separated steepwater at a temperature from 47.8°C (118°F) to 51.7°C (125°F) for between 10 to 40 hours provides a steepwater having low reducing sugar content. This characteristic renders the steepwater commercially attractive. Moreover, the yields of starch are high, providing another advantage in that starch yields frequently suffer when fermentation quality steepwater is produced. Finally, the steepwater is evaporated to provide a commercial product.

[0012] In preferred embodiments of this invention, the pH is adjusted at a time between 8 hours and 16 hours after the fresh steepwater is introduced.

[0013] While the pH adjustment can be carried out in the course of the steepwater process, in another embodiment of the invention the steepwater can be withdrawn from the process, the pH adjusted, and the steepwater returned to the process.

[0014] The following Examples will serve to illustrate the practice of this invention.

EXAMPLE 1

[0015] Steeping of clean yellow dent no. 2 corn was carried out in an industrial plant at different conditions as described below in two identical sets of steeps silos (A and B battery). One of these batterys is shown in Fig. 1 and comprises

[0016] Eight silos A1 to A8 of stainless steel with a filtering base B, of 67,800 gallons total volume and each diameter equal to 18' equipped with:

a piping 11 connecting the base of a given silo to the head of the silo itself, and to the heads of the other seven silos via a common drain pipe C2 and a common fill pipe C3 ensuring the recycling of the water into the silo itself or to any other silo, this piping also serving to withdraw the soaking water at a given time to a holding tank (not shown) through common pipelines shown in Fig. 1,
a bottom valve 14 for emptying the corn,
a tube and shell heat exchanger 16 with a circulating pump P-1 which controls the liquid flow through the heating tubes in heat exchanger 16 to the head of any other silo,
eight solenoid operated water drain valves V2 placed on pipeline 11 to segregate each silo from the common drain line C2 allowing water movement throughout the battery,
eight solenoid operated water fill valves V1 placed on pipeline 12 to segregate each silo from the common fill line C3 allowing water movement throughout the battery,
an outlet pipe C1 and valve V3 connecting to an incubation tank (not shown) that receives a batch draw of 2-4 gallons of water per bushel of corn ground with a capacity large enough to provide 24-40 hours of incubation time, connected to each silo through pipes C2 and 11, with valves V2 ensuring the segregation of water between the silos,
an inlet pipe 19 for SO_2 water, adjusted to 1000-2200 ppm, from which the SO_2 water is added as a batch by opening valve V5 and V1, replacing drawn water,
an inlet pipe 18 for alkali addition, to adjust pH in a range 4.2-5.5, from which the alkali is added into C2 by opening V4 V_2 and V_1 of the same silo, and starting pump P-1; this allows pH adjustments of water in any silo during any part of the steeping cycle,
pH meter Z-1 for measuring the pH of the water, Z-1 closes V-4 when pH reaches the desired set point.

[0017] The silos are filled with corn M and process water is moved countercurrently through the steep silos with the oldest corn contacting the newest water. In the system the corn does not move from steep silo to steep silo; only the water is advanced between silos. After 24 to 40 hours of incubation, the steepwater is moved to an evaporator (not shown) and is evaporated to 50% dry matter under a vacuum, at a temperature below 85°C.

[0018] The two identical sets of silos were operated at steeping temperatures of 47.8°C - 51.7°C (118° F - 125°F),

SO_2 concentrations of 1000 to 2200 ppm and steepwater draw rates of 7.6-15.1 litres (2.0 to 4.0 gallons) per 35.2 litre (bushel). B - battery was the control sample and A - battery was pH adjusted during steeping at designated times. A 100-gram sample of clean yellow dent no 2 corn was sealed in a 1.2" by 1.2" cheesecloth and placed into each silo in each battery after the silo is filled with corn. The corn samples in the cheesecloth were exposed to steep conditions identical to the corn steeped in each battery. After designated times during the steeping cycle, samples were pulled from each battery, cooled to stop further steeping, and tested for millibility according to the procedure described in Cereal Chem. 70:732-727.

[0019] The steepwater pH was adjusted to 4.6 in A-battery at various points during the steeping cycle as indicated in Table I while B - battery was maintained at normal pH conditions of 3.8 to 4.1 pH. This adjustment was made with the addition of 30% aqueous NaOH. The point in the steeping cycle where the pH was adjusted varied from 8 hours after the SO_2 water is introduced to the steeping process through inlet pipe 18 to 1 hour before the final steepwater draw.

[0020] Table I shows the results for comparisons of starch yields between the B - battery control samples and A-battery samples at various times during the steeping process. The graph of this data in Fig. 2 shows the relationship between pH adjust steep times and it's impact on starch yields. It should be noted that the most effective point of pH adjustment occurred between 8 to 16 hours after the SO_2 water is introduced to the steeping process with a second adjustment made 1 hour before the movement of the water to the incubation tank.

TABLE I

Starch Yield in % by Weight of Corn					
Steep Time (Hours*)	(Control) 0 hours	8 hours	12 hours	16 hours	18 hours
12	-	59.48	62.31	61.37	-
16	59.46	62.25	64.92	63.93	62.56
20	63.21	62.18	66.18	64.64	62.93
24	64.60	64.63	67.01	65.16	60.83
28	65.95	-	68.15	-	-

* Time after the SO_2 water is introduced to steeps.

EXAMPLE 2

[0021] One-gallon samples were obtained from the final steepwater draw from A-Battery while pH adjustments were occurring 8 hours after SO_2 water was introduced to the steeps. 200 mL aliquots were taken from the samples and pH adjusted to 4.9 to 5.0 and incubated at various temperatures ranging from 43.3°C (110 degrees F) to 55°C (131 degrees F) in the lab. Reducing sugar content was tested during incubation using the Corn Industries Research Foundation Division of Corn Refiners Association, Inc. Standard Analytical Method. Sixth Edition. D-52, J-58. Washington. This method measures percent by weight d.s. (dry substance) of reducing sugars in the samples.

[0022] Results shown in Fig. 3 indicate the reduction of the reducing sugars in the steepwater during the duration of incubation cycle. Samples maintained at or lower than 46.1°C (115 degrees F) during incubation began to exhibit sensory attributes of alcohol fermentation, which is detrimental to the process of producing fermentation grade steep-water. In addition, results of incubation temperatures above 50.6°C (123 degrees F) showed that a sufficient reduction in reducing sugars was not achieved. Therefore, the optimum temperature limits based on these results for reducing the amount of reducing sugars in steepwater during incubation would be from 47.8°C (118 degrees F) to 50.6°C (123 degrees F).

EXAMPLE 3

[0023] Additional reducing sugars tests were performed in the process to confirm lab data. Table II and Fig. 4 indicate resulting data of actual reducing sugars content before any pH adjusting occurred in the actual steeping process. Aliquots were withdrawn over a period of 3 months and 6 days. Consistently the percentage by weight d.s. reducing sugars was above 10 percent and averaged above 12 percent. These levels are well above the acceptable levels generally required for effective fermentation media. Table III and Fig. 5 indicate the actual percent reducing sugars after pH adjustment to 4.5-4.8 pH were made in the steep process 8-16 hours after the SO_2 water is introduced and with a second pH adjustment made to the steepwater 1 hour before movement of the water to incubation. Steepwater temperatures in all processes were 47.8°C - 50.6°C (118-123 degrees F) Aliquots were withdrawn over a period of 5 months. The results indicate that the steepwater had a reducing sugars content of an average below 5 percent by weight d.s.

TABLE II

Incubation Tank #3 Before pH Adjustment in the Steeps			
Day	%DE	Draw/Bu.	SO_2
1	11.6	2.64	
2	12.1	2.4	
3	15.5	2.99	
4	12.8	2.52	
5	14.8	2.16	
6	11.1	1.76	
7	11.6	2.26	
8	13.8	2.13	
9	17.3	1.88	
10	14	2.67	
11	13.3	3.32	
12	12.9	2.9	1500
13	11.3	3.03	1300
14	11.6	2.86	1550
15	11.8	2.96	1243
16	11.1	2.42	1526
17	11.8	2.4	1424
18	9.8	1.83	1475
19	10.1	1.85	1340
20	11.2	2	1425
21	13.2	2.72	1285
22	15.4	1.92	1281
23	13	2.55	1490
24	14.6	2.53	1450
25	9.4	1.62	1700
26	14.8	2.72	1900
27	14.2	2.38	1694
28	11.6	2.17	1725
29	14.9	2.51	
30	12.6	2.41	
Average	12.77	2.42	1488.71

TABLE III

Incubation Tank #3 After pH Adjustment in the Steeps			
Day	%DE	Draw/Bu.	SO_2
1	4.3	3	1255
2	5.2	3.19	1540
3	6.9	2.85	1615
4	3.3	3.41	1727
5	5.7	3.4	1600
6	4.6	2.8	1588
7	4.6	2.45	1841
8	4.7	2.88	1891
9	4.5	2.62	1620
10	6.1	2.8	1050

TABLE III (continued)

Incubation Tank #3 After pH Adjustment in the Steeps			
Day	%DE	Draw/Bu.	SO_2
11	5.4	3.2	1534
12	4.1	3.5	1520
13	5.1	2.43	1554
14	3.9	2.35	1555
15	4.2	2.69	*1546*
16	4.2	2.2	1394
17	4.5	2.9	1416
18	4.6	2.8	1610
19	4.5	2.7	1285
20	2.8	2.98	1522
21	4.8	2.43	1296
22	3.9	2.78	1560
23	3.8	2.86	1400
24	5.4	2.79	1462
25	6	2.7	1638
26	5.8	2.43	1730
27	6.5	2.14	1650
28	6.4	2.59	1736
29	5.8	2.56	1784
30	6.5	2.9	1006
31	5.1	2.46	1140
32	3.2	3.12	1186
33	3.6	2.28	1300
34	3.7	2.33	1280
35	2.9	2.54	1244
36	4.3	2.41	1128
37	4.3	2.36	1670
38	4.5	2.43	1650
39	6.8	2.56	1829
40	6.7	2.88	1700
41	6.6	2.13	1735
42	6.1	2.47	1720
43	5.4	2.37	1680
44	4.4	2.45	1850
45	4.3	2.18	1600
46	2.9	2.42	1300
47	3.1	2.47	1350
48	4.6	2.13	1400
Average	4.80	2.65	1513.69

Claims

1. A corn steeping process for producing corn steepwater having low reducing sugar content comprising

(a) introducing fresh steepwater containing from 1000 to 2200 parts per million sulfur dioxide to corn;
(b) steeping the corn for a steeping time greater than 10 hours at a temperature from 47.8°C (118°F) to 51.7°C (125°F);

(c) separating the steepwater from the corn at a rate of 7.6-15.1 litres (2 to 4 gallons) per 35.2 litre (bushel) of corn;
(d) adjusting the pH to from 4.2 to 5.5 at a time between 4 hours after the fresh steepwater is introduced to 1 hour before the separation of the steepwater from the corn ;
(e) maintaining the separated steepwater at a temperature from about 47.8°C (118°F) to about 51.7 °C (125°F) for between 10 to 40 hours; and
(f) evaporating the corn steepwater.

2. The process of claim 1 wherein the pH is adjusted at a time between 8 hours and 16 hours after the fresh steepwater is introduced.

3. The process of claim 1 wherein the steepwater is withdrawn from the process prior to adjusting the pH, the pH is adjusted and the steepwater having an adjusted pH is returned to the process.

4. The process of claim 3 wherein the pH is adjusted at a time between 8 hours and 16 hours after the fresh steepwater is introduced.

5. The process of claim 1 wherein the corn is steeped for a steeping time between 20 to 48 hours.

6. The process of claim 5 wherein the pH is adjusted at a time between 8 hours and 16 hours after the fresh steepwater is introduced.

7. The process of claim 5 wherein the steepwater is withdrawn from the process prior to adjusting the pH, the pH is adjusted and the steepwater having an adjusted pH is returned to the process.

8. The process of claim 7 wherein the pH is adjusted at a time between 8 hours and 16 hours after the fresh steepwater is introduced.

Patentansprüche

1. Maiseinweichverfahren zur Erzeugung von Maiseinweichwasser mit einem niedrigen Gehalt an reduzierendem Zucker, das umfasst;

(a) Einführen von frischem Einweichwasser, das 1000 bis 2200 parts per million Schwefeldioxid enthält, zu Mais;
(b) Einweichen des Mais mit einer Einweichdauer von mehr als 10 h bei einer Temperatur von 47.8 °C (118 °F) bis 51.7 °C (125 °F);
(c) Abtrennen des Einweichwassers vom Mais mit einer Rate von 7,6-15,1 Liter (2-4 Gallonen) pro 35,2 Liter (Bushel) Mais;
(d) Einstellen des pH-Werts auf 4,2-5,5 zu einem Zeitpunkt zwischen 4 h nach dem Einführen des frischen Einweichwassers bis 1 h vor dem Abtrennen des Einweichwassers vom Mais;
(e) Halten des abgetrennten Einweichwassers bei einer Temperatur von etwa 47.8 °C (118 °F) bis etwa 51.7 °C (125 °F) während zwischen 10 und 40 h; und
(f) Eindampfen des Maiseinweichwassers.

2. Verfahren nach Anspruch 1, wobei der pH-Wert zu einem Zeitpunkt zwischen 8 h und 16 h nach dem Einführen des frischen Einweichwassers eingestellt wird.

3. Verfahren nach Anspruch 1, wobei das Einweichwasser vor dem Einstellen des pH-Werts aus dem Verfahren abgezogen wird, der pH-Wert eingestellt wird und das Einweichwasser mit einem eingestellten pH-Wert in das Verfahren zurückgeführt wird.

4. Verfahren nach Anspruch 3, wobei der pH-Wert zu einem zeitpunkt zwischen 8 h und 16 h nach dem Einführen des frischen Einweichwassers eingestellt wird.

5. Verfahren nach Anspruch 1, wobei der Mais mit einer Einweichdauer zwischen 20 und 48 h eingeweicht wird.

6. Verfahren nach Anspruch 5, wobei der pH-Wert zu einem Zeitpunkt zwischen 8 h und 16 h nach dem Einführen von frischem Einweichwasser eingestellt wird.

7. Verfahren nach Anspruch 5, wobei das Einweichwasser vor dem Einstellen des pH-Werts aus dem Verfahren abgezogen wird, der pH-Wert eingestellt wird und das Einweichwasser mit einem eingestellten pH-Wert in das Verfahren zurückgeführt wird.

8. Verfahren nach Anspruch 7, wobei der pH-Wert zu einem Zeitpunkt zwischen 8 h und 16 h nach dem Einführen des frischen Einweichwassers eingestellt wird.

Revendications

1. Procédé de macération du maïs pour produire de l'eau de trempage de maïs possédant une faible teneur en sucre réducteur comprenant

   (a) l'introduction d'eau de trempage fraîche contenant 1000 à 2200 parties par million de dioxyde de soufre dans le maïs ;
   (b) la macération du maïs pendant une durée de macération supérieure à 10 heures à une température de 47,8°C (118°F) à 51,7°C (125°F). ;
   (c) la séparation de l'eau de trempage du maïs à un débit de 7,5 à 15,1 litres (2 à 4 gallons) pour 35,2 litres (un boisseau) de maïs ;
   (d) l'ajustement du pH de 4,2 à 5,5 à un moment situé entre 4 heures après que l'eau de trempage fraîche ait été introduite et 1 heure avant la séparation de l'eau de trempage du maïs ;
   (e) le maintien de l'eau de trempage séparée à une température d'environ 47,8°C (118°F) à environ 51,7°C (125°F) pendant 10 à 40 heures ; et
   (f) l'évaporation de l'eau de trempage du maïs.

2. Procédé selon la revendication 1, dans lequel le pH est ajusté à un moment situé entre 8 heures et 16 heures après que l'eau de trempage fraîche ait été introduite.

3. Procédé selon la revendication 1, dans lequel l'eau de trempage est retirée du processus avant d'ajuster le pH, le pH est ajusté et l'eau de trempage possédant un pH ajusté est renvoyée vers le processus.

4. Procédé selon la revendication 3, dans lequel le pH est ajusté à un moment situé entre 8 heures et 16 heures après que l'eau de trempage fraîche ait été introduite.

5. Procédé selon la revendication 1, dans lequel le maïs est macéré pendant une durée de macération située entre 20 et 48 heures.

6. Procédé selon la revendication 5, dans lequel le pH est ajusté à un moment situé entre 8 heures et 16 heures après que l'eau de trempage fraîche ait été introduite.

7. Procédé selon la revendication 5, dans lequel l'eau de trempage est retirée du processus avant d'ajuster le pH, le pH est ajusté et l'eau de trempage possédant un pH ajusté est renvoyée vers le processus.

8. Procédé selon la revendication 7, dans lequel le pH est ajusté à un moment situé entre 8 heures et 16 heures après que l'eau de trempage fraîche ait été introduite.

FIG. 1

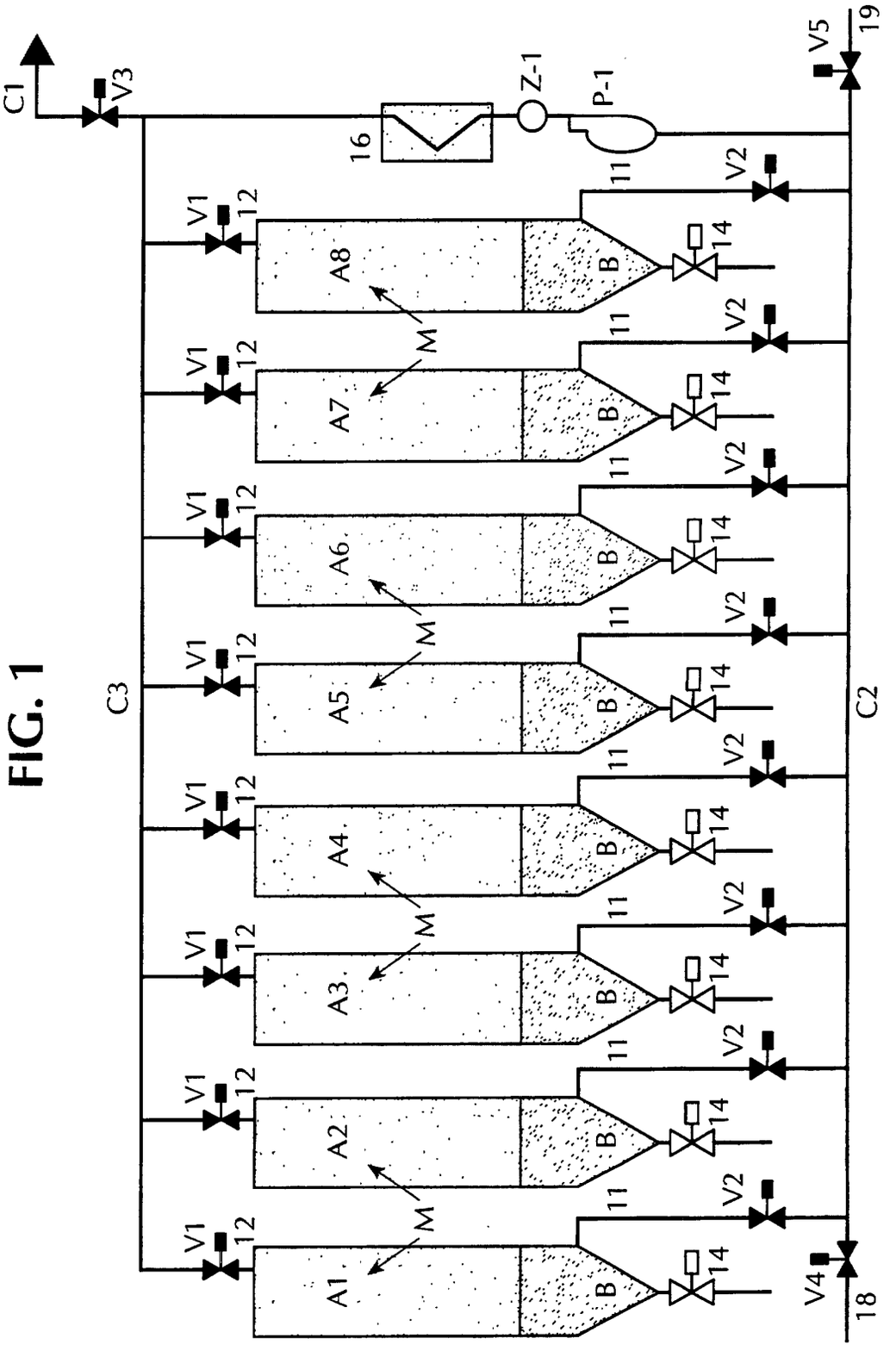

C1
V3
16
Z-1
P-1
V5
19
V1
12
V2
11
A8
A7
A6
A5
A4
A3
A2
A1
M
B
14
C3
C2
V4
18

FIG. 2

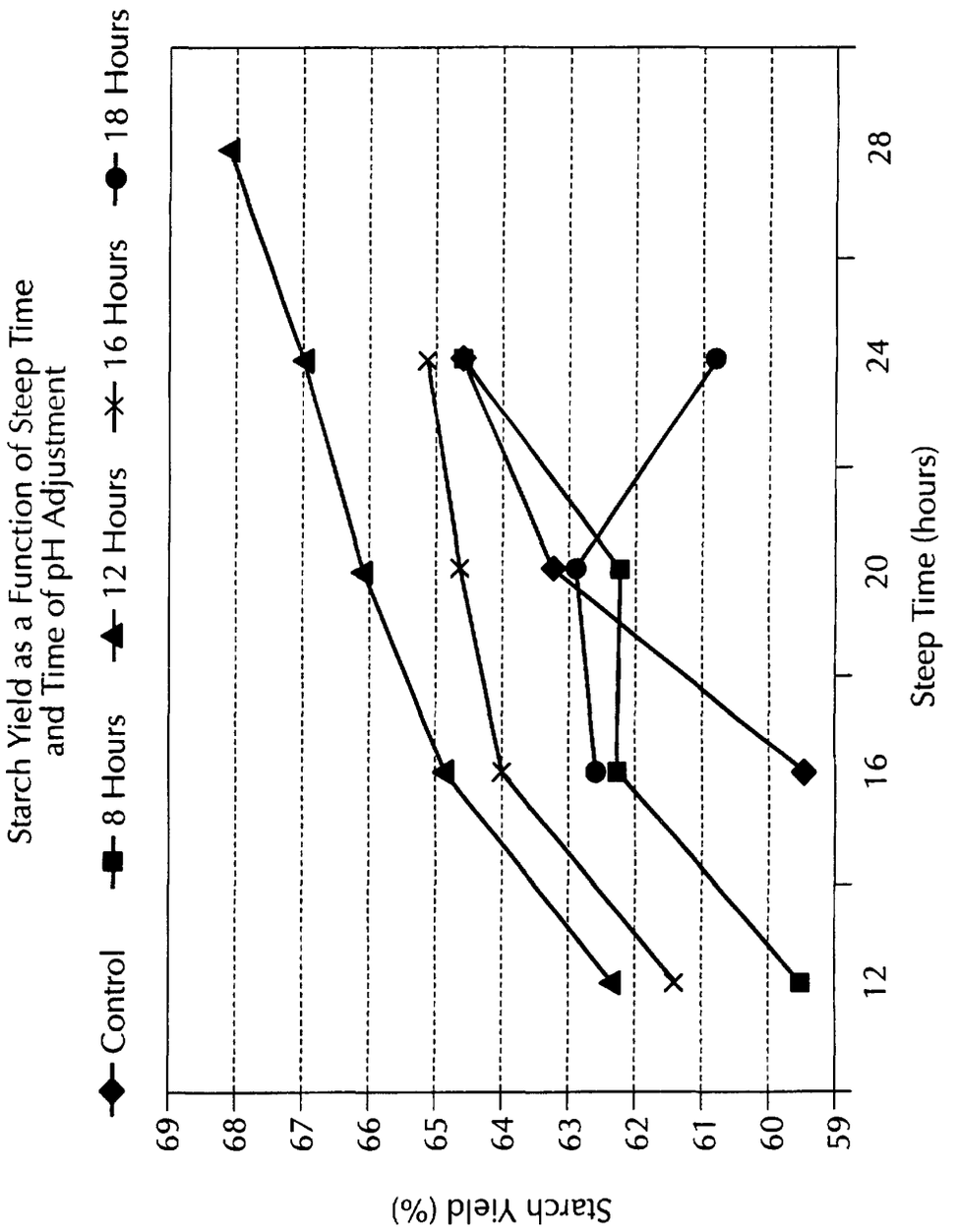

Starch Yield as a Function of Steep Time and Time of pH Adjustment
Control
8 Hours
12 Hours
16 Hours
18 Hours
Starch Yield (%)
69
68
67
66
65
64
63
62
61
60
59
12
16
20
24
28
Steep Time (hours)

FIG. 3

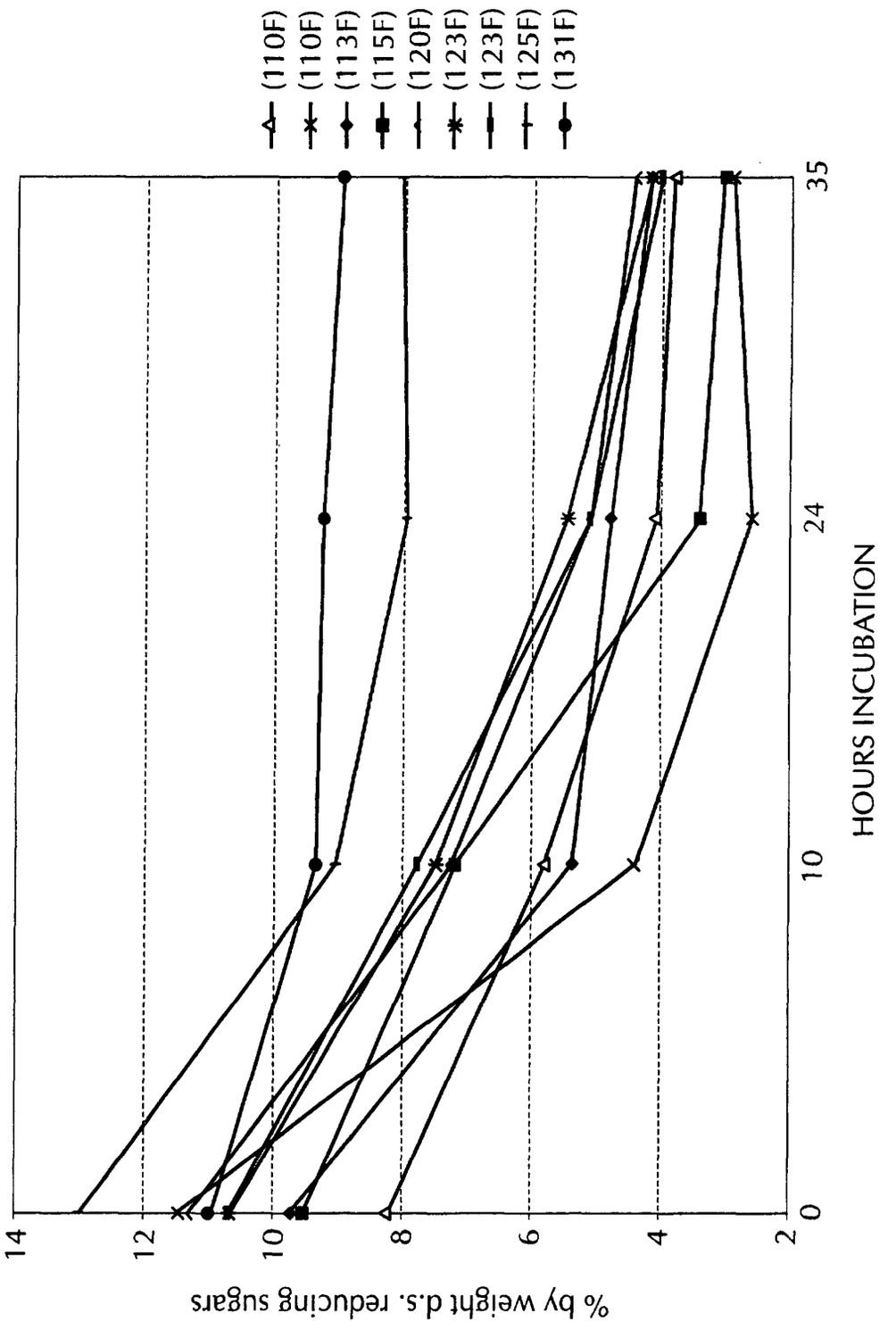

(110F)
(110F)
(113F)
(115F)
(120F)
(123F)
(123F)
(125F)
(131F)
% by weight d.s. reducing sugars
14
12
10
8
6
4
2
0
10
24
35
HOURS INCUBATION

FIG. 4

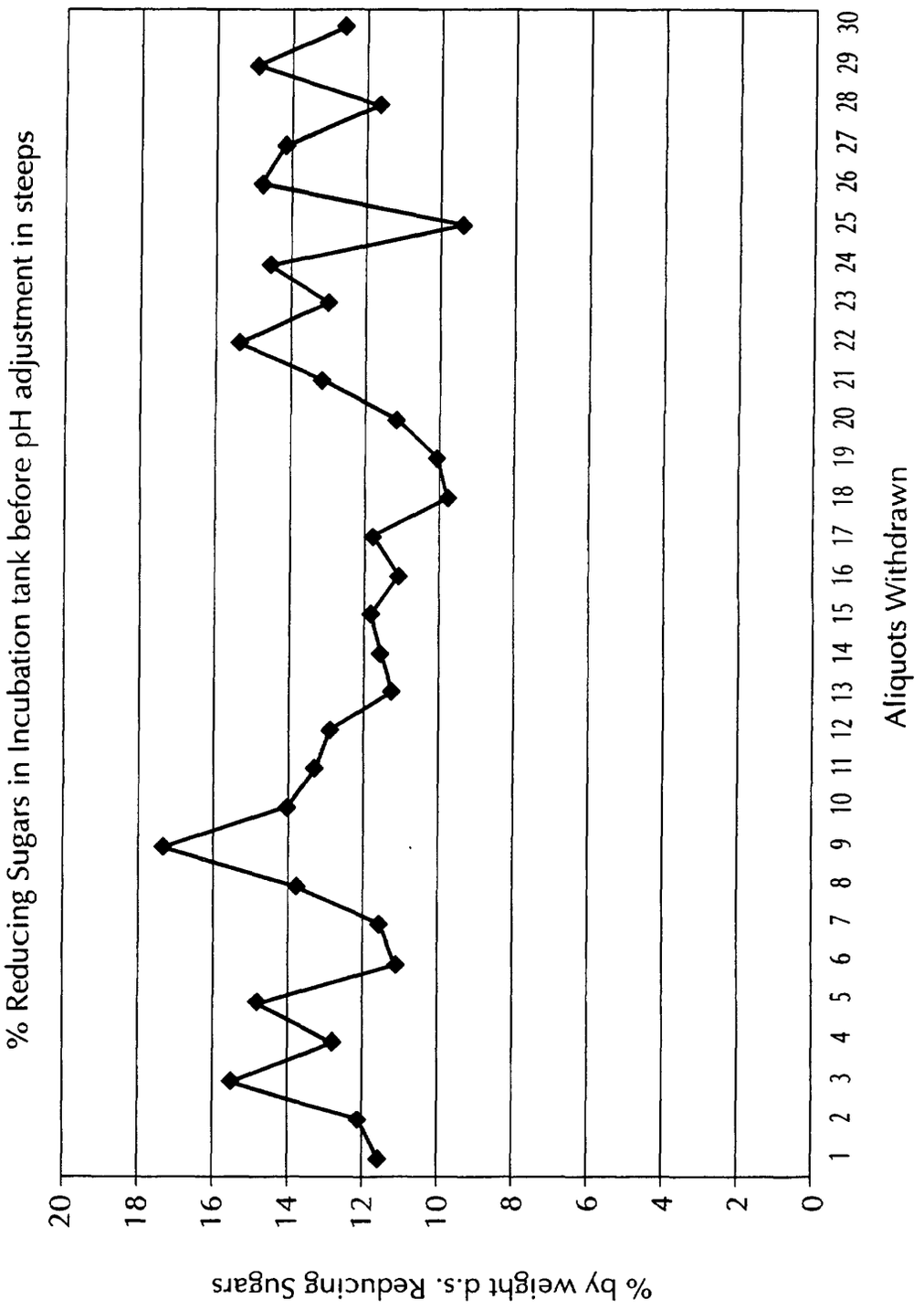
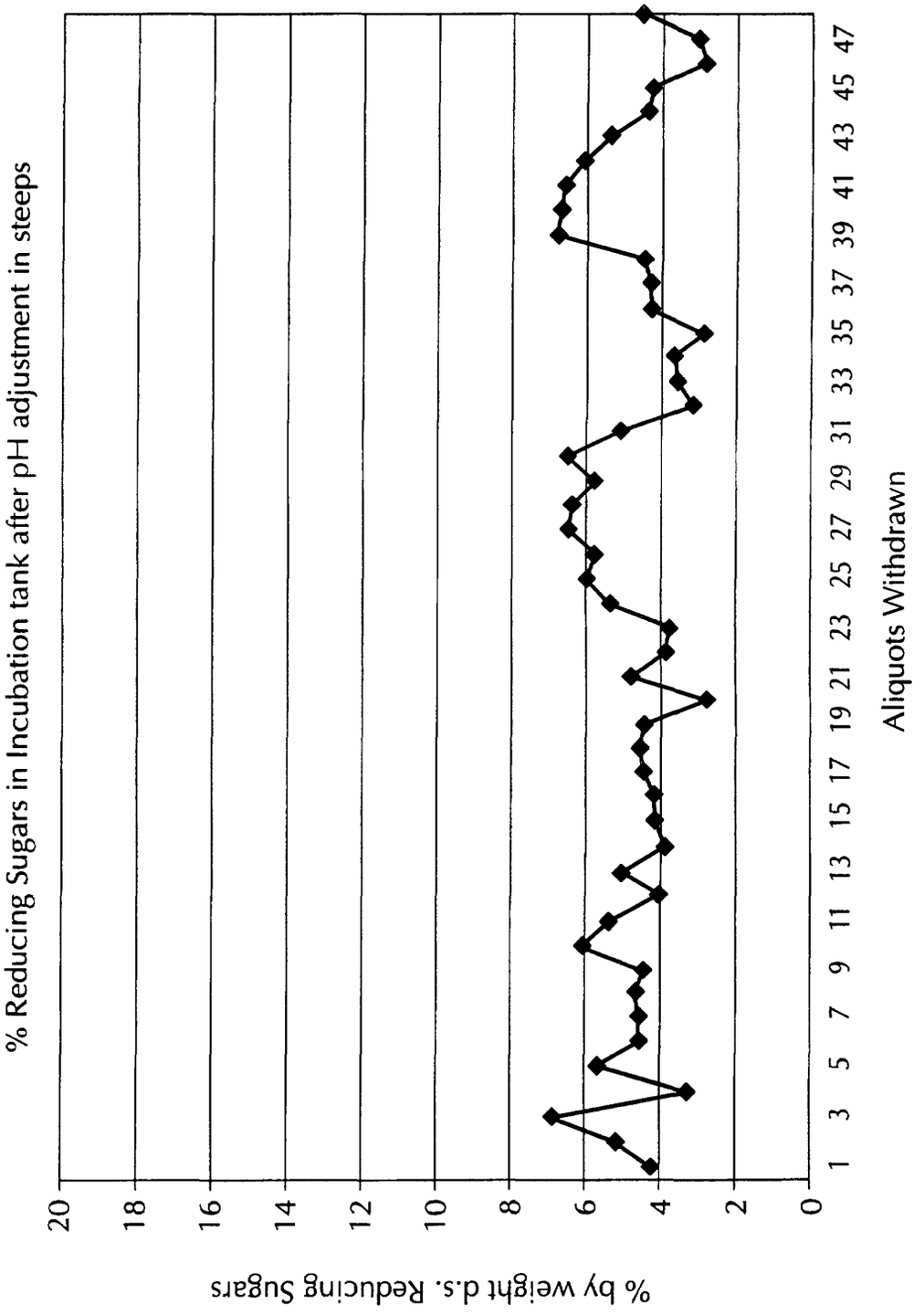

% Reducing Sugars in Incubation tank before pH adjustment in steeps
% by weight d.s. Reducing Sugars
0
2
4
6
8
10
12
14
16
18
20
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30
Aliquots Withdrawn

FIG. 5


% Reducing Sugars in Incubation tank after pH adjustment in steeps
% by weight d.s. Reducing Sugars
0
2
4
6
8
10
12
14
16
18
20
1 3 5 7 9 11 13 15 17 19 21 23 25 27 29 31 33 35 37 39 41 43 45 47
Aliquots Withdrawn