SORGHUM SIRUP MANUFACTURING TESTS

Including the Use of Clay Clarification And An Evaluation of Tested Varieties

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The various phases of work associated with the growing of sweet sorghums and the production or sorghum sirups have been under study for the past 10 years at the Oklahoma Agricultural Experiment Station. This bulletin contains a summary of the sirup-making tests, a brief report concerning the varieties that have been tested, and a summary of experiments dealing with modifications of existing sirup-making processes.

Instructions for the construction and operation of a sirup-manufacturing layout are available in the following bulletins from other experiment stations: "Farm Production of Sorgo Sirup," (11)* and "Growing Sorghum and Making Sorghum Sirup." (3)

Sirup-making Tests

The cane used in all of these tests was grown on creek bottom land classified as Yahola sandy loam. Rows were spaced 42 inches apart, with the stalks averaging six inches apart in the rows. Clean cultivation was practiced throughout. At harvest time, samples were secured from 100 feet of each row. This furnished a relatively uniform sample and a reasonable area from which to calculate yields.

In taking samples, the canes were stripped and headed in the field and immediately brought to the mill for weighing and processing. Juice was pressed from the canes in a small-sized commercial, power-driven mill, thus closely simulating commercial conditions.

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* Numbers in parentheses refer to the Literature Cited section, page 15.

During the first few years, samples were harvested in the mornings and the juice was processed the same day. In later years, the samples were cut and pressed in the late afternoon, the juice standing overnight in a cold room (35° F.) before being processed the next morning.

All juice processing was conducted under laboratory conditions. The evaporators used were small-scale pans holding five gallons of juice. The pans were made from heavy copper sheets and were modeled after the one developed by Walton and Ventre. (5) They were heated by three natural gas burners permitting accurate adjustments of the temperatures. (See Fig. 1.) Clay treatment of the juices was conducted in five-gallon pyrex bottles. This allowed close observation of the treated samples and measurement of the sediment layer.

CLAY TREATMENT OF JUICE FOR CLARIFICATION

Previous Experiments

The use of clay in the clarification of sorghum juice is not new, although it is not commonly used. H. W. Wiley (9) reported extensive experiments using clay as a clarifying agent; however, his emphasis was upon the use of clay and lime together, and with this combination, he found that many clays could be used. Willaman, *et al.* (10) stated that clay had been used in clarification, but that a satisfactory clay was difficult to find in Minnesota and they did not recomment its use. The most comprehensive recent report on clay clarification is that of Gaessler, *et al.* (1,2) which discusses the qualities necessary in a clay to secure good clarification. The authors found that an improved sirup resulted from the use of clay, although the yield of sirup was somewhat reduced.

Treatment Procedure at This Station

The first experiments at this Station were directed toward the use of clay in the cold state without the addition of lime. The results did not justify the efforts involved, although improvement was noted in the ease of sirup making. Heating the juice to improve clarification was then tried with good results.

After examining various modifications in procedure, the experimental one described hereafter was accepted as being best for laboratory procedures.

The raw juice was strained through several layers of cheesecloth into the evaporating pan and quickly brought to boil. The blanket of foam on top was pulled off and the juice, still near boiling, was drained into five-gallon pyrex bottles. A slurry of clay was added immediately to the hot juice, and the sample was agitated thoroughly by blowing air through it. (See Fig. 2.) If flocculation did not occur, more of the slurry was added and the sample again agitated. This process was repeated until flocculation or breaking occurred.

The sample was next allowed to stand from two to four hours. The top clear layer was then siphoned off and returned to the pan for concentration. Evaporation was continued until a temperature of from 224 to 226°F. was reached, when the final low flames were removed. After

Figure 1

the froth was skimmed off, the sirup was drained into jars and weighed to calculate the yield.

No definite amount of clay suspension was added to the juice. Rather, the slurry was continually added until the flocculating point was reached. The reason for this is that different amounts of clay are needed to clear the juice of different sorghum varieties and even for the same variety at different stages of maturity.

Juice from a few varieties was hard to clear, and even when cleared, the volume of sediment was too large to make the clearing operation

Figure 2

practical. However, settling was quite rapid in most instances, leaving the top layer of liquid a clear red or grey-brown color.

Samples harvested too early often were hard to clear and usually yielded an excessive volume of sediment. This difficulty was not experienced with any of the recommended varieties (4) that were harvested at the optimum stage.

Tests conducted by adding the clay slurry at varying temperatures showed that the nearer the juice was to boiling, the more satisfactory was the clarification. When the temperature fell below about 180°F., clumping was poor and settling was slow, often not being complete in a reasonable length of time.

The clay used in the tests was a maroon-colored clay shale from the Permian Red Beds. It was selected as being most efficient for laboratory purposes. A mechanical analysis of this clay gave the following results: sand—26 percent, silt—21 percent, 5 micron clay—53 percent, and 2 micron clay—48.5 percent.** The clay had a pH of about 7.45. These values seemed to be the most favorable when compared to other clays which were tested. Also, they conformed with the results of workers previously mentioned (2) in that some large particles were necessary along with the clay particles for quick settling.

The settling time was too long when pure clay was used. Clays of pH 5 to 6, and over 8, proved unsatisfactory for quick settling. It would seem that any clay that approximates the values of the clay selected for use in this experiment would prove satisfactory. Certainly, it should be possible by analysis and by trial to select a clay that would prove satisfactory and that could be found in or near a given locality.

The Advantage of Clay Treatment

All the variety tests were conducted using clay clarification. Organoleptic tests conducted by Station members have never indicated unfavorable effects on sirup that has been treated with clay. Generally, the treated samples were judged superior to the nontreated ones.

The greatest advantage of clay treatment lies in the reduced amount of skimming. In the tests, a properly treated sample required very little if any skimming.

One difficulty encountered with many samples was clabbering (or

** The analysis was conducted by William L. Garman, formerly Assistant Professor of Agronomy (Soils).

jelling) of the finished product upon standing. The clay treatment reduced this fault, but did not entirely eliminate it.

Clay Treatment on a Farm Scale

The use of clay for clarification as conducted in the laboratory proved both practical and desirable. The extension of this process to a farm scale, however, presents many problems. If clay of a desired type is not available nearby, there may be a transportation problem. This probably is not too serious since a commercial sirup-manufacturing plant found that a washtub full of clay was sufficient to clear 400 to 600 gallons of juice even if the clay was not re-used.

Previous workers (1,2) have stated that the clay may be re-used once, thus reducing mechanical losses. However, the workers at this Station do not favor such action unless the settlings are quite voluminous.

The real problem involved in the use of clay is the necessity of having settling tanks, and the effort required to transfer the heated juice to and from such tanks. An ideal situation would be a hillside layout with a heating pan on the upper side, a gravity flow to storage tanks or barrels where the clay could be added for settling, and then a gravity flow to the evaporators. The heating pan would not need to be overly large, since it could be emptied frequently. Since the juice would be pasteurized by boiling, the settling could be conducted safely overnight, thus giving a longer settling time.

Alternative set-ups could be incorporated in different situations. Perhaps one pan could do two operations—heat the juice, and then concentrate it after it had been settled in nearby tanks.

OTHER EXPERIMENTS WITH SIRUP

Centrifugation or Filtering

Some experiments were conducted in which the juice, after clarification, was run through a cream separator. This treatment removed some sediment from the treated juice; however, organoleptic tests of the finished product and observations of sirup behavior did not indicate sufficient results to justify the treatment. The use of a commercial filter press might prove valuable at this point; however, such presses are quite expensive and are not practical on a farm scale.

Jelling

Using the semi-sirup procedure suggested by Walton and Ventre, (6) several commercial diastatic enzymes were tried in an attempt to reduce jelling. Diastatic enzymes break down starches to sugars, thus removing one factor that causes jelling. Inconsistent results were obtained: clabbering was reduced in some samples, but not eliminated and little improvement was noted in others.

Clay treatment generally yielded better results than the trials with enzymes. Also, it is not felt that the results justify the use of enzymes on a farm-sized installation because of the labor and time involved in the process (cooling the semi-sirup, allowing it to stand with the enzymes, and then re-heating). However, enzyme treatment might prove valuable for those varieties that clabber badly.

There was a large varietal difference in respect to clabbering and jelling. However, none of the recommended varieties, with the exception of Sugar Drip, have so far proved unreasonably bad in this respect.

Crystallization

The use of invertase, which hydrolyzes table sugar to the simple sugars—glucose and fructose—has been recommended as an aid in preventing crystallization in sirup. (6) Such a use is generally effective, especially if a variety selected contains mostly sucrose. In the trials at this Station, most persons that tested the samples thought invertase-treated samples were slightly inferior in quality. This, along with the cost and extra labor involved—the semi-sirup must be allowed to stand for a time with the enzyme would seem to prevent its widespread use. An alternative to the use of invertase would be the selection of varieties that are relatively low in sucrose as compared to simple sugars. Data about such varieties are available in the publication by Webster, *et al.* (8) Leoti is the only one of the recommended varieties whose sirup is liable to crystallize.

RELATION OF HARVEST TIME TO SIRUP YIELDS

Time of harvest is more of a problem in Oklahoma than farther north because most varieties of cane here begin to mature in late summer, long before killing frosts. A variety study was conducted to determine the composition of the juice and the yield of sugar per acre in relation to time of harvest, and these data appear in an article prepared for publication. (7) A summary of the data indicates three points worth mentioning here:

(a) The maximum yield of sugar per acre, and presumably of sirup, will be secured at Stillwater if the crop is planted early and harvested during the general period of August 20 to September 20.

(b) There is less danger of crystallization at the earlier harvest times insomuch as there is a tendency for most varieties to increase relatively their percentages of sucrose as they mature, thus increasing the danger of crystallization in the sirup.

(c) All generalizations about harvest time are subject to changes in the weather, which may radically alter the desirable harvest time and ultimately the yields. For example, in one year when copious late rains fell, maximum yields were secured for many varieties—and particularly the late-maturing ones—at the last harvest time before frost, which was October 10.

Harvesting too early yielded a juice which was difficult to clear and gave a sirup possessing a green or immature taste. This is in agreement with reports of previous workers. (6) Station workers were able to make a good quality sirup from most varieties as late as October 10, which is contrary to some reports. Data are not available for sirup-making after this date.

Summary of Variety Tests, 1943-1952

In the variety tests, the major objective was to obtain information on both the agronomic and chemical sirup-making qualities of the more commonly grown sweet sorghum varieties. The following information was secured for each of the varieties studied: maturity date, standing ability, yield of juice and sirup, quality of sirup, total sugar content, and sugar ratios in juice and sirup and their reaction to environmental influences of drought, insects and diseases.

Reports have been published (4,8) covering shorter periods of time for certain of the more promising varieties grown in Oklahoma. This summary will serve to supplement these earlier reports. Other varieties have been tested at Stillwater and rejected for one reason or another. This account will serve to record briefly the results with such varieties, and to extend the reports already published on the recommended varieties. (4)

VARIETIES TESTED

African Millet.—A medium-maturing variety widely grown for forage in western Oklahoma which was included in the sirup tests for two years. Testing was discontinued because of exceedingly low yields and poor quality sirup. The average sirup yield for the two years was approximately 50 gallons per acre. Also, difficulties were encountered in processing the juice.

Collier.—A medium-early maturing variety included every year in the test. Highly variable sirup yields were obtained, ranging from 45 to 156 gallons per acre. The juice is exceptionally high in sugar, and for this reason, Collier is being utilized as a parent to breed new strains with this high sugar factor. It was used throughout the tests as the check variety for sugar content. Occasionally, sirup made from Collier has been of good quality; however, it seemingly carries some inherent characters which generally cause the sirup to scorch before the desired density is reached. In addition to these difficulties, the sugar ratio of the sirup is consistently high on the sucrose side (as much as 9 to 1) which can result in crystallization when the sirup is stored.

Colman Y.—A medium-early variety introduced from California in 1946, and included in the experiments since that time. Sirup yields were highly variable; however, under favorable weather conditions, it produced between 100 and 150 gallons per acre of high quality sirup. It was used as the principal variety to check the quality of sirup from other varieties and new strains. It is highly susceptible to foliar diseases, and because of this fault, the variety has been removed from the recommended list. Research is under way to develop a foliar disease-resistant type of Colman Y.

C. P. Special.—A late-maturing type introduced in 1944 from the Federal Sugar Station, Meridian, Mississippi. The original lot of seed produced a wide variation of medium-late to late-maturing plants. From this mixed lot of plants, a medium-late strain was selected and purified. This selection was tested for two years, and it yielded around 150 gallons of fair to medium quality sirup per acre. The selection was highly susceptible to lodging from dry weather and to chinch bugs, and therefore was removed from the test.

Crystal Drip.—An early variety obtained in 1949 from the Keller's Seed Store in Shawnee, Oklahoma. The original seed when grown produced numerous off-type plants. The variety was purified by bagging the seed head through a series of years. Production of sirup has ranged from 80 to 175 gallons per acre. Appearance and quality of the sirup has rated good to excellent. A final evaluation of this variety is not possible because of the wide fluctuation in yields and the short testing period. The name Crystal Drip was given the variety in 1952 by the Keller Seed Store.

Gooseneck.—Two fixed selections were made from a mixed lot of Gooseneck seed. Selection No. 1 is characterized by black glumes, and Selection No. 2 is characterized by brown glumes. Testing of these selections was started in 1945. Selection No. 1 was discarded after three years because of low yields and poor quality sirup. Selection No. 2 has been retained in the test to date with yields ranging from 80 to 170 gallons per acre of good quality sirup. At times, this selection has showed the same lodging defects found in Selection No. 1. This weakness will probably eliminate it from becoming a recommended variety.

Honey.—A late-maturing variety included for the full duration of the tests. It has consistently been a high producer of excellent quality sirup. In only one year did it produce less than 100 gallons of sirup per acre, and in its best year it produced 220 gallons per acre. It has a minor defect of producing numerous tiller stalks which are immature when the main stalk is ready to harvest. Some susceptibility to drought and chinch bugs has been noted. When environmental conditions are favorable, Honey is a reliable and satisfactory sirup variety.

Bug Honey.—An early-maturing selection made at the Experiment Station in 1944 from a planting of regular Honey. It closely resembles Honey in plant and head characters, but matures about two weeks earlier and is more resistant to chinch bugs. Testing of the selection began in 1945 and was continued through 1950. Sirup yields were generally below 100 gallons per acre, and in the majority of years the quality was poor. Even though this selection has high chinch bug resistance, the sirup prepared from it is of such poor quality that the variety cannot be recommended.

Iceberg.—A medium-late variety obtained from the Federal Sugar Station in 1944 and included in the tests since then and tested yearly for sirup-making qualities. Sirup yields have been around 150 gallons per acre, with maximum production exceeding 200 gallons per acre. It has consistently made an excellent quality sirup. The major defect of the variety is its sensitiveness to dry, hot weather, and because of this weakness, the stalks have lodged even in medium dry seasons. The variety shows promise in localities where ideal weather prevails throughout the growing season.

Kansas Orange.—A medium-maturing variety selected and named by the Kansas Experiment Station. The variety was tested in the sirup trials for a four-year period. It was eliminated from the test because of a stalk-lodging defect and a low sirup-yielding potential. An average of the sirup yields for the four years was slightly below 100 gallons per acre. The sirup made from this variety graded good to excellent.

Leoti.—An early-maturing variety included in the test each year. The sirup yields indicate that this variety will consistently yield approximately 100 gallons of sirup per acre. Because of its early-maturing habit, this yield can be expected even in dry seasons when production and sirup quality is reduced in later-maturing varieties. The sirup made from Leoti always rated good to high in quality. Some years the sirup, upon standing, showed crystal formation. This apparently can be attributed to a high ratio of sucrose to simple sugars. Because of the sureness of production and the ease with which the juice processes, Leoti has been recommended as a commercial and farm sirup variety for all of Oklahoma. (4)

Red X.—A medium-maturing variety included for the entire test period. Sirup yield per acre fluctuated from 65 gallons in the poorest season to 160 gallons in the most favorable one. The average production was around 100 gallons per acre. Sirup quality ranged from average to below average. This variety was never quite good enough to recommend nor quite poor enough to discard.

Sugar Drip.—A medium-late variety used throughout the test. Its high yielding capacity was used to check sirup production of other varieties. Yields of the variety ranged from 140 to 210 gallons per acre, with production exceeding 150 gallons most of the years. Generally, an acceptable sirup was made from this variety; however, in some years the sirup clabbered badly and possessed an objectionable flavor. These undesirable features can be minimized by clay treatment of the juice or by careful processing. The variety is widely adaptable and resistant to lodging, and it possesses a high yielding capacity. Thus, it is recommended for both sirup and forage production in all of Oklahoma except for the Panhandle and the northwestern counties.

Sumac No. 1712.—A medium-late maturing selection of Sumac sorgo made at the Woodward Field Station. The variety was used principally to check the sirup-making qualities of the Sumac type of sweet sorghums. It ranked high in sirup-yielding capacity, averaging around 150 gallons per acre for the testing period. The sirup did not crpstallize on standing, but is was generally strong flavored, and it was always liquid. Quality scorings on the sirup usually placed it average or below. This selection of Sumac appears better suited for forage purposes than for sirup making.

White African.—A medium-late maturing variety included in all the test years because of its high sirup-yielding potential. Yields of sirup were around 175 gallons per acre. However, the sirup was always dark, strong flavored, and it often clabbered on standing. Because of these defects, the variety is not recommended.

BREEDING PROGRAM

The preliminary studies described in the opening paragraphs of Part II indicated that several of the varieties possessed desirable sirup-making qualities; however, none of the varieties was ideally suited for this purpose. Each showed certain strong characteristics, as well as some weak characteristics, for sirup production. No two of the varieties seemed to carry the desirable and undesirable factors in equal proportions.

With this information at hand, it seemed believeable that improvement in sirup sorghums would be possible by making various crossing combinations with the better varieties. Then, by a selecting and testing program, it should be possible to obtain new types carrying more of the desirable characteristics than the parent lines.

A breeding program was initiated in 1945 using the following varieties for the parent lines: Leoti, Collier, Colman Y, Sugar Drip, and Honey. Testing of selections from these various combinations started in 1948 and has continued to date. Of the voluminous amount of material tested the past five years, only two selections have shown enough improved qualities to warrant consideration. These are tan-plant types from a Collier-Leoti cross. They possess many desirable characteristics. However, at times the sirup made from them has been somewhat inferior in quality, and frequently crystals will form when the sirup is allowed to stand. Both selections have been re-combined with Colman Y, Sugar Drip, and Honey in an attempt to improve these defects. Fourth generation selections from these new combinations are available for checking their sirup-making qualities in 1953.

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