CURING AND FERMENTATION OF CIGAR LEAF TOBACCO.

INTRODUCTION.

The production of tobacco adapted to the different market demands has become a prominent factor in national economy. Of particular importance is the production of superior cigar leaf tobacco. The filler leaf of a cigar must above all things have a good flavor, good aroma, and good burn. In the wrapper leaf, however, still other qualities come in, such as elasticity, pliability, size, shape, color, size of the veins, the fineness and peculiar grain of the Havana type, and the smooth silkiness of the Sumatra.

Little is known of the chemical properties of the leaf, especially of those which contribute to the flavor and aroma. It is probable that the actual amount of nicotine is relatively unimportant, and it is certain that the excellence of the leaf and its adaptation to market demands is not dependent, except in a very general way, upon the amount of nicotine. It has long been known that certain of the potassium salts, especially potassium chlorid, can not be used at all for the production of high types of cigar tobacco, as they give the leaf a poor burn. It is furthermore an old experience of tobacco growers that excessive nitrogenous manuring tends to produce a large leaf, of inferior quality, containing an increased amount of nicotine. If the prime object of tobacco culture were the production of nicotine, as the prime object in raising sugar beets is the production of sugar, then the rational procedure would be to furnish an excess of nitrogenous manures, but nicotine alone does not make a good cigar tobacco any more than alcohol alone would make a good wine. The substances producing the flavor and aroma, therefore, although probably present in minute quantities, are much more important than the actual percentage of nicotine found in the cured leaf.

Whitney[1] has shown that tobacco suited to our domestic cigars is grown only upon certain soils and under certain climatic conditions. It appears, therefore, that the leaf capable of being converted into a cigar leaf through the ordinary processes of curing and fermentation must

[1] Bull. No. 11, Division of Soils, U. S. Department of Agriculture.

possess certain characters. A fresh leaf has no specific taste, nor has it any specific odor, but the finished leaf has a sharp, saline taste and a characteristic odor.

From the time the tobacco leaf is gathered in the field until the manufacture of the cigar and even afterwards a series of highly interesting changes take place in the leaf, as a result of which the characters of the finished leaf are developed and fixed. There are three stages in these changes, viz, (1) the curing process; (2) the sweating, or fermentation; and (3) the cold sweat, after-fermentation, or aging, as it is variously called.

THE CURING.

There are two periods in the curing process: The first period, in which the cells of the leaves are still alive and induce processes of metabolism; and the second period, in which the cells have died and the chemical changes have therefore no connection with the living protoplasm. In the former period, which may last only a few days (longer with the ribs), the starch content is dissolved and the sugar formed is partly consumed by an increased respiration[1] and partly transported to the ribs, where, as Müller-Thurgau has shown, starch may be formed again. In the latter period the enzyms alone are active.

Decrease of protein.—With the consumption of a large amount of the sugar a state of inanition or starvation sets in, and the reserve protein is attacked by an enzym, trypsin-like in character, the action of which will continue after the death of the cells. A cold-prepared aqueous extract of a fresh leaf will show albumin on the addition of nitric acid and warming, while the cured leaf does not give this reaction. The reserve protein and a certain albuminous portion of the nucleo-proteids of the protoplasm will thus finally be split and transformed into amido compounds and bases, only the remaining nucleins resisting, hence the decrease of protein matter in the curing and fermentation process will stop at a certain point. Such proteolytic processes proceed not only in plants exposed to darkness, which means their starvation or inanition, but also in all cases where reserve protein must be dissolved to enable further development, as in germination or development from bulbs.

It is in full accordance with physiological principles that when cells are in want of nourishment they produce a larger amount of enzyms than when well nourished. This explains why tobacco leaves killed immediately after being gathered will show imperfections when after having been moistened they are subjected to the curing process. The enzyms that have been produced during the inanition state of the

[1] The respiration of a pile of such fresh leaves may soon lead to a considerable and even injurious rise of temperature, as in the respiration of germinating barley on the malting floor. A moderate rise is often intentionally brought on, as it hastens the curing. Sometimes this rise of temperature is called sweating, although the cause here is a different one from the true sweating, or fermentation, following r curing.

cells, however, will naturally remain active after the death of the protoplasm from starvation has set in.[1]

Considerable variation has been found in the total nitrogen content of the fresh leaves, as well as in the amido nitrogen content of the cured leaves. The amount of the former may vary in American, Greek, and German tobaccos from 2, 3, or 4 per cent to 8 per cent of the dry leaf. One-third of this and even more can turn into amido compounds in the curing process.

Regulation of heat and moisture.—Further changes, relating to color and flavor, set in with the death of the cells. However, it requires a most judicious regulation of the moisture, temperature, and ventilation of the barn where the tobacco leaves are hung up to obtain those changes which characterize cured tobacco of a superior quality. This curing process may last four weeks or even much longer.[2] When the weather is too dry all the chemical changes in the leaves come to a premature stop, but on the other hand when it is too moist the danger of mold development arises. In the former case the barns must be eventually kept closed and water introduced, while in the latter case careful application of heat may be resorted to.

An interesting experiment in curing by artificial heat has been described by E. H. Jenkins.[3] Some farmers have tried the burning of sulphur with the intention of killing the mold spores by sulphurous acid, but this requires the utmost precaution, as the leaves themselves might easily be injured and even all further action in them stopped.

Sometimes mold fungi will develop unnoticed in the stems, appearing distinctly later on, when the sweating operation has begun. All diseased leaves must be discarded before fermentation begins in order to avoid further damage by the spreading of the fungi. Tobacco growers in Florida recognize the white mold, the yellow mold, the blue mold, and the stem-rot mold, the latter being the worst and causing much damage. Sturgis has described a bacterium causing pole burn of tobacco,[4] and further determined the fungus causing the stem rot to be *Botrytis longibranchiata*.[5] Jenkins reports that the pole burn disease "may destroy a portion or even the whole of the harvested crop within forty-eight hours after the time when the trouble is first noticed."

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[1] It is somewhat difficult to prove the presence of diastase in healthy normal leaves, as very small quantities may resist extraction.

[2] The drying, or curing, for good cigar tobacco requires about as much time in America as it does in Europe. Tscherwatscheff, a Russian, has described the American method as requiring but four days with applications of artificial heat (Landw. Jahrb., 1875). What he had seen, however, was nothing but the preparation of light-colored cigarette tobacco as practiced in North Carolina, Virginia, and Kentucky. In curing cigar tobacco fire is resorted to only when damp, foggy weather prevails for a long time.

[3] Conn. Agr. Expt. Sta. Ann. Rept., 1897; *Ibid.*, 1892, p. 38.

[4] Conn. Agr. Expt. Sta. Ann. Rept., 1891.

[5] *Ibid.*, Sturgis's list of tobacco diseases.

Flavor.—The development of the flavor of cured tobacco has not yet been explained. At first a decided flavor of cucumbers[1] is generated, which later on is entirely replaced by the rank and common straw smell of cured tobacco, giving rise finally to the superior tobacco flavor developed by the sweating or fermentation process.

Color.—As regards the brown color of cured and fermented tobacco, there can hardly be any doubt that not only one but several compounds contribute by their chemical changes to its development. Of course the first supposition would be that the tannin, by being changed into a phlobaphene (a brown product), is the principal cause.[2] Thus, for example, in the autumn, when the leaves of oaks and of various other trees containing tannin die off, a brown coloration sets in. But the intensity of the brown color of the fermented tobacco leaf does not run parallel to the different concentration of the tannin in the cell systems of the leaf. A healthy tobacco leaf was placed with its base in a dilute solution of ferrous sulphate (about 1 per cent) for from twelve to fifteen hours, at the end of which time this reagent had risen to the tip of the leaf, thereby partly killing it. A reaction in the form of a black color appeared, principally in the epidermis and to some extent also in the mesophyll, but not at all in the vascular bundles. This black coloration seemed to be restricted to the chloroplasts.

The epidermis of cured leaves, however, contains the least amount of coloring matter and is sometimes entirely devoid of it, with the exception of the gland hairs, while the mesophyll cells always contain a brown substance in irregular-shaped or rounded masses. The principal part of the brown matter, however, is in the veins of the leaves and even the most minute ramifications of the vascular bundles appear to be a much darker brown than the neighboring mesophyll cells. The circumstance that the veins contain less nicotine than the rest of the leaf also militates against the view that the coloring matter is principally due to the oxidation of nicotine. However, there occurs in the veins a bitter principle that does not seem to occur in the rest of the leaf, and perhaps this may contribute to the color.

It is easy to show that several compounds contribute to the brown coloration in well-cured leaves. In the first place, much brown matter is extracted by cold water. Leaves thus exhausted will yield up another portion of brown matter to warm, dilute sulphuric acid, and finally still another portion[3] of a different chemical behavior is extracted by a warm, dilute solution of potassium hydrate.

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[1] The expressed juice of a fresh tobacco leaf is at first without odor, but it gradually assumes that of fresh cucumbers, which later on is destroyed by putrefaction.

[2] According to Savery, the tannin of tobacco is identical with that of coffee. There exists, evidently, several kinds of phlobaphene, depending on the kind of tannin.

[3] This latter portion is a mixture of several compounds, some colorless and pectose-like, and one colored and phlobaphene-like. Twenty-five grams of fermented tobacco from Florida yielded 0.51 grams of this product. The cell membranes of the tobacco thus treated exhibit under the microscope a swollen appearance.

One author has assumed that the chlorophyll is first attacked in the curing process and destroyed, but this is not correct. The green color of the chlorophyll is in the beginning merely covered by the brown substances. In the thin samples of fermented leaves of a light-brown color green spots may frequently be noticed, and even dark-colored, freshly fermented leaves may sometimes yield a greenish solution upon extraction with strong alcohol. It is of some interest to note that the brown matters are insoluble in absolute alcohol.

Ammonia.—An interesting feature in the curing and fermenting process is the formation of a small amount of ammonia. As the green leaves contain some asparagin, the formation of ammonia might be due to a small extent to the decomposition of this amide, which readily yields ammonia and aspartic acid. But in certain tobacco crops there occur only minute quantities of asparagin. Certain amido compounds formed by decomposition of proteids and also a part of the nicotine in decomposing probably yield the principal amount of ammonia. The nicotine undergoes, in the fermentation process at least, a considerable diminution, as explained below.

The opinion that the ammonia deteriorates the quality of the product is certainly unfounded, as Fesca has correctly pointed out. It has been demonstrated by Behrens that during the curing process a part of the sulphur of the decomposed proteids is oxidized to sulphuric acid and that the amount of compounds soluble in ether decreases. The latter consist of a fatty substance and a volatile oil of disagreeable odor derived principally from the gland hairs.

The total loss of dry matter in the curing process is subject to great variation, depending mainly upon the amount of starch present at the time of gathering, as above stated. The diminution of dry matter may be as much as 40 per cent.

The principal changes in the curing process may be summed up as follows:

(1) Disappearance of starch.
(2) Formation of sugar and its partial disappearance by respiration.
(3) Decomposition of protein with formation of amido compounds.
(4) Decrease of fatty matter.
(5) Decrease of tannin.
(6) Change of color and flavor.

THE SWEATING OR FERMENTATION PROCESS.

The so-called fermentation process develops in the tobacco leaves the characteristic qualities of the commercial article. It is natural to suppose that the same agency which finishes the curing process after the death of the cells remains active during the so-called fermentation process also. The fermentation follows immediately after the curing when both are done by the grower, but where the cured tobacco is bought up by manufacturers several months may pass before it is subjected to the sweating process. This operation begins when the tobacco is in the proper "order" or "case," being brought into this condition naturally on a damp day, or by an exceedingly cautious moistening, avoiding any visible water on the leaves. The amount of water applied must just suffice to bring on moderate imbibition. The total amount of water necessary to bring on a normal sweat is from 18 to 25 per cent of the moistened leaf. A portion of this water (about one-fourth) is again lost during the sweat.

The sweating of the Florida leaf in bulk requires from six to eight weeks, the original crude and rank smell of the cured tobacco being gradually changed to the proper aroma of the finished tobacco, and the glossy appearance and the texture[1] being well brought out. Light-colored wrappers require a slower and cooler fermentation than the dark-colored leaves used as dark wrappers or fillers.

Rise of temperature.—When the cured tobacco is sold by the farmer a large number of leaves are tied together at the base, forming "hands." At the beginning of the sweat such "hands" are well shaken in order to open all the foliage and admit air to every part. Then commences the moistening, when necessary, which is done by exposing the "hands," under continuous shaking, to a current of steam issuing from a pipe; by spraying with a fine spray; or by dipping, in which case the bases of the "hands" are plunged into water and shaken, the adhering water being soon drawn by capillary attraction into the leaf. These "hands" are then packed, with the butts outside, in piles 4 to 5 feet wide and 12 to 15 feet long. The rooms, which contain a large number of such piles, are kept warm, and steam passes freely from a number of pipes into the air of these rooms to secure uniform moisture, as otherwise the warming piles would soon become too dry. The temperature of these piles rises in from one to two days considerably above the temperature of the fermenting room and may reach 52° C. (126° F.) or higher. Repacking becomes necessary in from three to four days in order to check the rise in temperature and to shake out the leaves. The lower "hands" are now placed on the top and the outer ones in the center in order to give all leaves an equal chance to improve. The temperature now rises more and more slowly, the next repacking not being necessary before about seven or eight days. Altogether the piles are repacked from five to eight times. When the temperature rises too high the color or the aroma may be injured, hence frequent examinations are necessary. These examinations are made by pushing the hand into the piles, a decision being reached by the sense of feeling.[2]

Oxidation.—Tobacco manufacturers are well aware of the fact that a moderate quantity of air should gain access to the interior of the

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[1] The texture, or grain, of the leaf means to tobacco manufacturers small points plainly visible on the extended leaf. It appears that these points are the bases of the gland hairs, most of which break off in the curing and sweating processes.

[2] For details relative to the treatment of the fermenting tobacco heaps the reader is referred to Farmers' Bulletin No. 60 and to the next report on tobacco.

fermenting piles and that undue pressure must be avoided in order not to diminish this access of air more than is necessary to insure an accumulation of heat. Not only are numerous little channels left naturally in the piles, but diffusion also will set in as soon as the air in the piles becomes warmer than the surrounding atmosphere.

Repeated efforts have been made to replace the sweating or fermentation process by a direct oxidation. Dr. Mew, of the Army Medical Museum of this city, assures the writer that some experiments made by him about twenty years ago to improve the cured tobacco leaf by direct application of a dilute solution of permanganate of potassium resulted in an essential improvement, the product being milder. Similar results have been recently mentioned by Kiessling.[1] In Germany a patent has been granted to the firm of Siemens & Halske for treating tobacco with ozone. However, oxidation often takes quite an undesirable turn, and the danger of destroying the aroma is quite as great, if not greater, than the likelihood of developing it by artificial means.

Losses.—Jenkins has shown[2] that the losses in fermentation are apparent in the nicotine, protein, amido compounds, nitrogen-free extract, and also, to a much less extent, in the ether extract. The loss of nicotine varies considerably in different samples and was found by Jenkins to range from one-sixth to one-half in three samples analyzed. Behrens observed in one sample a decrease of nicotine from 1.46 per cent in the cured leaf to 1.07 per cent in the fermented leaf. Dambergis found in air-dry Greek tobaccos, having from 7 to 14 per cent of water, from 2.8 to 0.7 per cent of nicotine.[3]

The question as to how much the loss of organic matter amounts to during the sweating process can be answered only approximately and by comparing parts of one and the same leaf, but a constant result will never be reached, as the nature of the proceeding in fermentation brings on differences in temperature, water content, and access of oxygen, and thus leads to variations. In the fermenting heap thick and thin leaves occur, often varying more than 20 per cent in weight for an equal surface area. Leaves grown in the shade are thinner than those exposed to direct sunlight, and in hot, dry summers the leaves are thicker and coarser than in moist, rainy seasons.[4] These conditions of course naturally influence the result.

Some tobacco manufacturers estimate the average loss during the fermentation process to be 15 per cent (organic matter and water together), while others estimate the loss of solid matter alone to be

[1] Der Tabak, Berlin, 1893.

[2] Conn. Agr. Expt. Sta. Ann. Rept., 1891.

[3] Oesterreich. Chem. Zeitg., No. 16, 1898.

[4] In the rainy season of 1891 Sumatra tobacco leaves weighed 52 grams per square meter, while in the dry season of 1892 the leaves grown on the same spot weighed 80 to 90 grams per square meter. Behrens explains this difference by the larger intramolecular spaces produced by excess of moisture (Landw. Vers. Stat., 1894, Band 43, p. 272).

from 4 to 5 per cent. According to Jenkins,[1] the losses may be even larger. He reports that "the upper leaves, short seconds, and first wrappers lost, respectively, by fermentation 9.7, 12.3, and 9.1 per cent of their total weight. But while three-fourths of the loss in the case of the short seconds consisted of water, in the case of the upper leaves almost three-fourths of the loss was dry matter. The first wrappers lost a little less dry matter than water."

Development of gases.—The formation of ammonia can be noticed by the characteristic odor in the fermenting rooms, but the amount is not so high as one might naturally be led to suppose from the intensity of the smell. About 3 liters of air from the interior of a fermenting pile when drawn through 25 cc. of Nessler's reagent, produced a light yellow color, indicating about 0.05 milligram of ammonia. No trace of hydrogen sulphide is given off. Test tubes containing filter paper moistened with basic lead acetate remained perfectly colorless for twenty-four hours in the fermenting heaps, hence it may be safely concluded that no protein decomposition resembling putrefaction takes place.[2] The amount of carbonic acid given off was also much smaller than would naturally be expected from the apparent energy of the action.

Starch.—Small quantities of starch are sometimes found in fermented tobacco when the curing process has not been carried out properly in all parts of the leaf or in parts of leaves broken, or injured by fungi, as observed by Müller-Thurgau, but this occurrence of a small percentage of starch interferes with the flavor just as little as does the closely related cellulose. The well-prepared tobacco wrappers from Florida examined by the writer did not show a trace of starch. The fact, however, that in the curing process the solution of the starch is going on with great energy forms a contrast to the observation that in certain cases remnants of starch remain unattacked during the fermentation process. This admits of hardly any doubt that the diastase[3] is gradually destroyed, perhaps by the proteolytic enzym.

Sugar.—As to the disappearance of the last remnant of sugar during the sweating, amounting, according to Müller-Thurgau, to from 1 to 3.3 per cent, some authors assume oxidation to carbon dioxide and water, and others assume a partial transformation to acetic acid. When it is taken into consideration that an alkaline medium can soon change glucose into organic acids (gluconic, saccharinic, etc.), especially in the presence of air, a more simple explanation would be at hand than

[1] Conn. Agr. Expt. Sta., Ann. Rept. 1892, p. 28. The leaves used for comparison were most carefully selected and were as nearly alike in color, size, and texture as possible.

[2] Nessler's comparison of the sweating process to putrefaction is certainly not admissible; neither is his declaration that the formation of ammonia is not normal, but simply a sign of true putrefaction.

[3] Diastase is absolutely necessary to dissolve and saccharify the starch. The dextrin and maltose thus formed may afterwards be transformed into glucose by the living protoplasm itself, wherever this latter comes under consideration.

the assumption of a perfect combustion of the glucose. There are organic acids present in the original tobacco leaf, such as citric, malic, and oxalic acids, in the form of neutral salts. A part of these acids may be changed and destroyed in the fermentation process, while other acids may be formed by the changes the glucose undergoes.[1] The nicotine is bound to organic acids and is not present in the free state; besides, most of the ammonia formed is in combination with organic acids, but a part of it is easily liberated by boiling the aqueous extract of the fermented tobacco. These vapors have a strong alkaline reaction and an ammoniacal odor, and are due either to the volatilization of some ammonium carbonate or to the dissociation of a neutral ammonium salt of a bibasic acid.

Tannin.—The amount of tannin, like that of nicotine, also decreases in fermentation. It varies from 0.3 to 2.3 per cent in commercial tobaccos. The Florida tobacco of 1898 contained only traces of tannin after the fermentation was over. The amount of fatty matter, or, more correctly speaking, of substances soluble in ether, was found by Jenkins[2] to decrease in fermentation from 3.5 to 2.8 per cent of the dry matter. Behrens observed in one case a decrease from 9.14 per cent in the cured to 8.34 per cent in the fermented leaf. The amount of such fatty substances was found to vary in different samples from 1.8 to 10 per cent and in some cases even more. The decrease of fatty matter during fermentation is probably due to the volatilization of a volatile ethereal oil. It is certainly very improbable that some true fat was oxidized to carbonic acid and water. Little attention has been given thus far to the small amount of resins in tobacco.

Fiber.—In regard to the fiber, Jenkins determined its amount in Connecticut tobacco as ranging from 13 to 14 per cent. Fesca and Jmai found the range in Japanese tobacco to be from 13 to 15 per cent. Only the ribs contained more—from 22 to 24 per cent.[3]

Ashes.—The amount of mineral matter is subject to very great variation, namely, from 10 to 27 per cent.

Nitrate.—A question of special interest is the fate of the nitric acid probably present exclusively as potassium nitrate in fresh leaves. Some authors believe that nitrification goes on during the fermentation process, which would lead to an increase of nitrate in the fermented leaf. This, however, has never been proved by chemical analysis and is indeed

[1] The precipitate obtained by copper acetate from a hot, aqueous extract of fermented tobacco contains, among other things, some succinic acid. The writer did not recognize butyric acid among the volatile acids in Florida tobacco, but acetic acid was present.

[2] Conn. Agr. Expt. Sta., Ann. Rept. 1890. Correct comparison is, however, possible only in calculating for a constant, e. g., cellulose.

[3] There are still certain substances in the fermented tobacco which thus far could not be characterized. Some analyses show from 1.7 to 18.9 per cent of nitrogenous extractive matter and from 8.6 to 16.7 per cent of indefinite insoluble matter. There exists great difficulty in isolating certain compounds from these mixtures.