A STUDY OF CERTAIN CONSTITUENTS OF THE LEAF AND THEIR RELATION TO THE BURNING QUALITIES OF TOBACCO[1,2]

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Introduction

The term "burning qualities" as applied to smoking tobacco, usually includes several different elements. Of these, according to GARNER (**9**, p. 8), the fire holding capacity, the evenness and completeness of combustion, and the character of the ash are of most importance. A knowledge of the factors influencing the burning qualities of cigar-leaf tobacco is of considerable importance, both from the standpoint of the producer and the manufacturer, as the value of tobacco for cigar manufacture is based almost wholly upon these qualities. For this reason, therefore, considerable investigational work has been conducted upon this subject, and, as a result, it is now known that several factors, especially those of a chemical nature, influence the "burn" to a considerable extent.

Review of previous investigations

SCHLÖSING (**23**) was one of the first investigators to study the chemical factors affecting the burning qualities of tobacco. He treated tobacco leaves with various salt solutions and found that potassium salts of various organic acids were decidedly beneficial. He attributed their effects to the tendency of these salts to swell on heating, thus exposing more surface of combustible material.

By impregnating filter-paper with solutions of various salts, NESSLER (**20**, p. 73) found that the sulphate and carbonate of potassium increased the fire-holding capacity to a marked degree. He also observed that potassium acetate promoted combustibility in a manner similar to the carbonate, although this salt does not possess the power to swell when heated, at least to a marked degree, therefore, SCHLÖSING's interpretation of the catalytic effect of potassium salts of organic acids was probably not correct.

In analyzing the ash of good-burning tobacco, VAN BEMMELEN (**24**) always found a greater quantity of potassium present as the carbonate and lesser quantities as the chloride and sulphate.

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[2] This investigation was conducted in cooperation with Dr. W. W. GARNER, of the U. S. Bureau of Plant Industry, Office of Plant Nutrition and Tobacco Investigations, and Professor F. D. GARDNER, Department of Agronomy of the Pennsylvania State College.

MAYER (**16**) studied the influence of organic and inorganic substances on the capacity of filter-paper to glow or burn with a flame. The results obtained with inorganic compounds were similar to those obtained by NESSLER. In addition, he found that nearly all organic substances promote burning with a flame.

It has been suggested by BARTH (**2**), that the burn and characteristic glow of a good tobacco is due to a reduction of the alkali salts to oxides and small quantities of free metal, and that such a reduction would increase the efficiency of these metals as oxidizing catalysts.

BEHRENS (**4**), as a result of his investigational work on the burning qualities of tobacco, states that a high content of potassium in combination with citric, malic and oxalic acids largely determines the burning quality. GARNER (**9**, p. 19), is also of the opinion that potassium in such combinations is highly desirable, and further suggests that the favorable action of the carbonate obtained from the combustion of these substances may be due to its effect of functioning as a catalyst in combustion by taking up carbon dioxide and forming the acid carbonate at the most favorable moment and losing it later on. KRAYBILL (**12**), however, obtained results which seem to disprove this theory.

RIDGWAY (**22**) found a striking relationship existing between the fire-holding capacity and the degree of aggregation of various salts normally present in tobacco leaves. Under certain conditions of curing, it appears that there is a noticeable tendency for calcium and magnesium to crystallize as malates, citrates and oxalates. As these bases do not appear to promote the fire-holding capacity to any considerable degree, such a process is desirable, as relatively larger quantities of the tissues are thus left free to burn. He found that where such a crystallization did not take place, the burning quality was always poor.

According to LOEW (**13**, p. 38), the more oxidizable material, and the more oxidation going on within the tobacco leaf, the more will the crystals or "grain" develop in the curing and sweating processes and hence it will in many cases, although by no means in all, confirm the idea of some tobacco manufacturers that a well-developed grain is a good sign of the quality of the tobacco.

That chlorine exerts a deleterious effect on the burning quality of cigar-leaf tobacco is quite definitely established. MAYER (**14, 15, 16**), BEHRENS (**4, 5**), NESSLER (**17, 18, 19, 20**), FESCA (**6**), GARNER (**9**, p. 18), AMES and BOLTZ (**1**, p. 191), OLSON (**21**, p. 11), JENKINS (**11**, p. 95), and others have reported on the poor quality of cigar-leaf tobacco fertilized by chlorine-containing material. JENKINS (**10**) also states that while some chlorine is absolutely essential for the development of the tobacco plant, a large excess may prove deleterious to the burn.

Some work has been done to show a relation between the water-, ether-, and alcohol-soluble constituents of the leaf to its burning quality. GARNER (9, p. 18), made water extractions and found that the extracted leaf lost its glowing capacity. On examining the extract, he found that it contained the chloride, sulphate, nitrate, malate and citrate of potassium, ammonium and nicotine, and small quantities of lime and magnesium. Extracts from both good-burning and poor-burning leaves contained about the same quantities of potassium but the inferior leaf contained a much higher percentage of mineral acids. Hence he concluded that the potassium salts of organic acids such as malic and citric are the chief factors controlling the burn. Extraction with alcohol had little or no effect on the burn. It has also been shown by GARNER (9, p. 8) that some of the weaker organic acids, on the order of tannic, in combination with nicotine, are deleterious to the burning quality.

GRAHAM and CARR (7) made petroleum ether, sulphuric ether, alcohol and hot water extractions of tobacco and showed a relation between poor-burning quality and a high extractive content. They showed that tobacco fertilized with sodium nitrate or muriate of potash yielded a high amount of extractives.

Purpose and plan of the experiment

The object of this investigation was to make a study of the chemical composition of a given strain of cigar-leaf tobacco[3] in relation to its burning qualities, particularly as regards its fire-holding capacity. Particular attention was to be given to the determination of the quantity of ether-soluble organic acids present in cured and fermented samples, and also to their form of combination, as measured by the water-soluble and insoluble alkalinity of the ash. In addition, the effects of chemical treatments and climatic conditions were to be given due consideration.

MATERIALS USED

Eighteen plants, carefully selected from each of ten plots, 1/30th of an acre in area, located at Ephrata, Lancaster County, Pennsylvania, were harvested at the end of the growing season in 1925 and removed to the curing shed, where they were air cured. These samples were taken from the shed at the end of the curing season and dried under laboratory conditions. When they were thoroughly dry, the leaves, stems and stalks were separated, finely ground and transferred to air-tight containers and kept for analysis. This same procedure was followed in 1926.

The chemical treatment of the plots from which the samples were taken is shown in table I.

[3] HIBSHMAN strain, Pennsylvania Broadleaf.

TABLE I

FERTILIZER TREATMENTS OF THE EXPERIMENTAL PLOTS

PLOT	TREATMENTS[a]										
	MANURE	SULPHATE OF POTASH	MURIATE OF POTASH	NITRATE OF POTASH	MANURE SALT	BONE FLOUR	PRECIPITATED BONE PHOSPHATE	ACID PHOSPHATE	UREA	AMMONIUM NITRATE	COTTON-SEED MEAL
	tons	lbs.	lbs.	lbs.	lbs.	lbs.	lbs.	lbs.	lbs.	lbs.	lbs.
A–1	10	....	200	....	....	250	....	....	......	......	400
A–2	10	200	....	....	....	....	....	300	......	......	500
A–5	10	....	....	....	....	....	....	....	......	......	....
A–6	10	200	....	....	....	....	....	300	......	......	500
A–9	10	....	....	....	400	250	....	....	......	......	400
A–10	10	....	200	....	....	....	....	300	......	......	500
B–1	10	....	200	....	....	....	....	300	65	......	....
B–2	10	....	....	214	....	....	120	....	......	......	....
B–3	10	200	....	....	....	....	....	300	65	......	....
B–6	10	200	....	....	....	....	....	300	......	85	....

[a] The potash, phosphoric acid and nitrogen-carrying materials, with the exception of manure and cottonseed meal constituted acre applications of approximately 100 lbs. of potash, 48 lbs. of phosphoric acid and 38 lbs. of nitrogen.

Methods of analysis

In determining the total organic acid content of the different samples of tobacco, 10 grams of the material were transferred to a porcelain evaporating dish and 15 cc. of a 20 per cent. H_2SO_4 solution added. The mixture was then stirred thoroughly by means of a spatula, and finally enough powdered pumice stone was added to make the mass appear fairly dry. The mixture was then placed in a paper thimble and extracted with anhydrous ethyl ether for 24 hours. To the ether extract was added 200 cc. of water, the ether allowed to evaporate, the solution cooled, filtered and made up to a volume of 250 cc. Aliquots of 25 cc. were then taken and titrated with 0.04 N. NaOH, using phenolphthalein as an indicator.

In determining the crude ash and alkalinity of the ash, 2 grams of the sample were transferred to a platinum dish and carefully charred, the residue treated with water and filtered. The insoluble portion was placed again in the dish and ignited to constant weight. The filtrate was next added to the dish, evaporated, and the total residue carefully heated, cooled and weighed. The weight of the residue was calculated as crude ash.

About 150 cc. of hot water were next added to the crude ash and filtered. The residue on the filter-paper was washed well and the filtrate obtained was titrated with N/10 H_2SO_4, using methyl orange or xylene cyanal methyl orange as indicator.

The residue on the filter-paper was then placed in a platinum dish, treated with an excess of N/10 H_2SO_4, and the solution heated to boiling and filtered. The excess acid was then titrated with NaOH standard solution and the insoluble alkalinity was calculated accordingly. The usual methods of analysis were employed to determine the remaining constituents.

Experimental

Experiment I

Samples of the 1925 crop were taken and the more important constituents of the web determined. The results obtained are given in table II.

Experiment II

The total ether-soluble organic acid content of the stems, stalks and web portions of the cured samples, and also of the leaves of the fermented samples were determined. In addition, the total water-soluble and insoluble alkalinity of the web portions of the cured material were likewise determined.

The number of cc. of N. NaOH required to neutralize the ether-soluble organic acids in 100 grams of the air-dry material, and the number

TABLE II

THE PERCENTAGE COMPOSITION OF THE WEB OF CURED TOBACCO LEAVES OF THE 1925 CROP AS INFLUENCED BY FERTILIZER TREATMENT[a]

PLOT	CRUDE ASH	SiO_2	P_2O_5	CaO	MgO	SO_3	K_2O	Cl	N	NICOTINE
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
A- 1	24.08	2.10	0.50	7.88	1.25	1.46	1.89	1.12	4.70	4.89
A- 2	23.79	1.88	0.40	7.06	0.96	1.49	1.94	0.12	4.33	5.14
A- 5	23.99	2.52	0.40	7.30	1.24	1.40	1.93	0.19	4.38	4.61
A- 6	22.78	2.23	0.43	7.58	1.32	1.14	1.65	0.17	4.20	5.16
A- 9	24.15	2.14	0.44	7.93	1.21	1.27	1.95	2.29	4.37	4.56
A-10	23.10	2.23	0.44	7.64	1.15	1.35	1.97	0.95	4.63	4.79
B- 1	24.45	3.53	0.45	7.08	1.16	1.29	1.84	1.26	4.73	4.51
B- 2	23.32	2.09	0.48	7.83	1.31	1.61	1.97	0.19	4.60	4.82
B- 3	23.34	2.22	0.42	7.30	1.08	1.45	1.93	0.22	4.67	4.90
B- 6	23.59	3.87	0.43	7.17	1.14	1.28	2.03	0.25	4.87	4.09

[a] All percentages based on moisture-free material.

of cc. of N. H_2SO_4 required to neutralize the alkalinity of the ash of 100 grams of the same material are given in table IV.

EXPERIMENT III

Samples of the 1926 crop were taken after curing and determinations made of crude ash, chlorine, lime, magnesia and potash content of the web portions. In addition, the alkalinity and ether-soluble organic acid con-

TABLE III

THE CHEMICAL COMPOSITION OF THE 1926 CROP OF TOBACCO

PLOT	CRUDE ASH	Cl	K_2O	CaO	MgO	ALKALINITY OF ASH			ETHER-SOLUBLE ORGANIC ACID CONTENT
						Sol.	Insol.	Total	
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	cc.	cc.	cc.	cc.
A- 1	18.03	0.94	2.13	6.39	0.94	36	293	329	305
A- 2	22.85	0.17	2.29	6.43	0.89	48	283	331	281
A- 5	23.79	0.24	2.55	6.44	0.75	34	278	312	290
A- 6	21.08	0.17	2.38	6.29	0.86	51	323	374	290
A- 9	23.34	1.95	2.69	6.56	0.82	17	333	350	274
A-10	22.80	0.95	2.33	6.79	1.06	37	326	363	293
B- 1	22.78	1.07	2.04	6.83	1.09	25	296	321	286
B- 2	22.92	0.21	2.13	6.79	0.95	40	320	360	293
B- 3	22.54	0.19	3.37	6.75	0.88	39	306	345	283
B- 6	20.70	0.21	2.19	6.78	1.40	32	320	352	300

tent were also determined on the same samples. The results are presented in table III.

TABLE IV

THE TOTAL ETHER-SOLUBLE ORGANIC ACID CONTENT AND ALKALINITY OF THE 1925 CROP OF TOBACCO

Sample	Plant part	Cured material: Alkalinity of ash			Cured material: Ether-soluble organic acid content	Fermented material: Ether-soluble organic acid content
		Sol.	Insol.	Total		
		cc.	cc.	cc.	cc.	cc.
A– 1	Web	11	343	354	333	332
A– 1	Stem				234	
A– 1	Stalk				100	
A– 2	Web	39	322	361	336	
A– 2	Stem				300	
A– 2	Stalk				107	
A– 5	Web	22	332	354	347	351
A– 5	Stem				327	
A– 5	Stalk				139	
A– 6	Web	13	343	356	351	345
A– 6	Stem				304	345
A– 6	Stalk				128	
A– 9	Web	8	315	323	314	325
A– 9	Stem				167	
A– 9	Stalk				95	
A–10	Web	13	232	336	331	316
A–10	Stem				223	
A–10	Stalk				106	
B– 1	Web	14	317	331	313	305
B– 1	Stem				225	
B– 1	Stalk				103	
B– 2	Web	19	353	372	362	341
B– 2	Stem				311	
B– 2	Stalk				118	
B– 3	Web	35	309	344	345	324
B– 3	Stem				319	
B– 3	Stalk				163	
B– 6	Web	19	321	340	333	317
B– 6	Stem				324	
B– 6	Stalk				137	

Experiment IV

Samples of the web portions of the cured leaves of the 1925 and 1926 crops were taken and their hydrogen-ion concentration determined by the quinhydrone method (**3**). The results obtained are given in table V.

TABLE V

The hyrrogen-ion concentration of the 1925 and 1926 crops of tobacco

Year	Plots									
	A–1	A–2	A–5	A–6	A–9	A–10	B–1	B–2	B–3	B–6
	pH	pH	pH	pH	pH	pH	pH	pH	pH	pH
1925	5.96	5.96	5.78	5.76	5.97	6.01	5.38	5.74	5.34	5.61
1926	5.95	6.19	6.08	6.16	6.10	6.20	6.37	6.78	6.08	6.27

Experiment V

Burning tests were conducted on strips of the fermented leaves of the 1925 and 1926 crops of tobacco. These strip tests of the fermented tobacco are shown in table VI.

TABLE VI

Strip tests[a] of the 1925 and 1926 crops of tobacco after fermentation

Plot	Burn 1925	Burn 1926
A– 1	Poor	Poor
A– 2	Fair	Fair to good
A– 5	Poor	Poor
A– 6	Good	Good to fine
A– 9	Poor	Poor
A–10	Poor	Poor
B– 1	Poor	Poor
B– 2	Good	Good to fine
B– 3	Fair to good	Good to fine
B– 6	Fair	Fair to good

[a] Definitions of terms in strip tests:

Very poor = tobacco does not hold fire. Coals badly.
Poor = tobacco holds fire but a few seconds. Coals badly.
Fair = tobacco holds fire over five seconds. Coals moderately.
Good = tobacco holds fire evenly over ten seconds. Coals slightly.
Fine = tobacco holds fire evenly until consumed. Coals very slightly.
Excellent = tobacco burns evenly until consumed without coaling and with light colored ash.

WEATHER CONDITIONS

Certain observations were made as to the weather conditions which prevailed during the growing seasons of 1925 and 1926. In 1925 the temperature was relatively high from May 31st until June 10th, and no precipitation occurred until June 25th. July was marked by severe electrical storms and heavy washing rains. The temperature rose gradually during the first two weeks of the month of August. No rain occurred from August 15th to September 13th. The plants were harvested shortly after this period.

The weather conditions for 1926 were quite different. It was moist and cool until June 10th, after which time the temperature rose to a relatively high degree and severe storms occurred during this hot period. The hot weather continued through July and August, but rain storms were quite frequent, breaking all previous rain records for August, and the excessive heat lasted until the plants were harvested. Following are the figures showing the actual precipitation during the growing season of 1926.

MONTH	RAINFALL inches
June	4.31
July	4.57
August	9.12
September	4.47

No accurate measurement of the rainfall during the growing period was made in 1925, but, on the whole, the precipitation was much less than for the season of 1926.

Discussion of results

Experiment I, table II, shows that the chemical composition of the 1925 crop, from an elemental standpoint, was rather constant regardless of the fertilizer treatment. More chlorine, however, was found in the plants receiving chlorine-bearing compounds. The water-soluble and insoluble alkalinity of the ash also varied to a certain extent as shown by experiment II, table IV. The form of potash used appeared to play a part in causing this variation. Where the plants received muriate of potash, the water-soluble alkalinity was less as a rule than where the sulphate was used. A higher total alkalinity was obtained where potassium nitrate was applied. This table also shows a very wide potassium-calcium ratio, a wider ratio than is commonly found in good burning tobacco.

Analyses of the crop of 1926, according to experiment III, table III, show that approximately the same quantity of chlorine was present as in the 1925 crop. On the other hand, the lime and magnesia contents were lower and the potassium content was higher in the 1926 samples, but the potassium-calcium ratio was still too wide for satisfactory combustion. Where chlorine was present in relatively large quantities, a low water-soluble alkalinity of the ash was again apparent. On the whole, however, the water-soluble alkalinity of the 1926 crop was higher than that of the 1925 crop. The principal results obtained from these investigations show, with few exceptions, that there is a correlation between the water-soluble alkalinity of the samples and their burning qualities, and that this effect is more pronounced than that of the relative quantity of ether-soluble organic acids. It appears evident also that the form and amount of potash, and to a certain extent the season, have a great deal to do with the total quantity of water-soluble alkalinity present. If this is true, it is important to account for these effects.

Owing to the limited number of samples studied, it would not be proper to draw many definite conclusions, but it appears that these samples contain too little potassium and too great a quantity of calcium for the most desirable combustion. The data also indicate that the sulphate of potash is more desirable as a carrier of potassium than is the muriate. If potassium functions normally within the plant, it must act as a base. Combined with chlorine or sulphur it does not have the desirable property of neutralizing organic acids to any considerable extent. As chlorine is not normally required in any large quantities by the tobacco plant, it would probably tend to remain in combination with potassium, at least to a certain extent. On the other hand, sulphur may be utilized in relatively large quantities, and under these conditions more potassium would be rendered available for the neutralization of organic acids. As a matter of fact, however, analyses of the plants showed that the sulphur content was not increased by the addition of sulphates, so the reason for the difference in the basic properties of potassium from the sulphate, as compared with the muriate, must be sought elsewhere.

It is a well-known fact that the principal replaceable base of soil colloids, in a calcareous district such as Lancaster County, Pennsylvania, is calcium, and that the addition of potash salts to such a soil should lead to the replacement of calcium from such combinations and the formation of calcium salts. At the same time, it is possible to reverse this process. In other words, there is a competition existing between calcium and potassium, whereby either one or the other goes into colloidal combination. Under laboratory conditions, a sample of soil may be treated with muriate of potash and the replaced calcium may be completely removed by leaching, leaving potassium behind in basic form. On the other hand, it is quite possible to remove all absorbed potassium by the use of a solution of calcium chloride, leaving calcium behind in colloid combination and in basic form. Under natural conditions, neither of the above effects is ever observed, but a relatively dry year, such as in 1925, would have a different effect on the availability of potassium than would a comparatively wet year, as in 1926. Continual leaching of the soil would have the effect of rendering potassium available for absorption in basic form by the tobacco plants, while such a condition would not hold true under relatively dry conditions where soluble calcium salts tend to accumulate in the upper soil layers. Under the former conditions the tendency would be, in the light of our present knowledge, for potassium to assume the carbonate form. Under the latter condition, potassium would tend to assume the form of the muriate or sulphate, as the case may be. These reasons would tend to explain, in a measure, the beneficial effects of a wet season on the burning qualities of Lancaster County cigar-leaf tobacco, but would not necessarily hold true to any appreciable extent on soils lacking in colloidal material, unless the presence of relatively large quantities of calcium tends to inhibit the maximum absorption of potassium by plants.

The beneficial effect of sulphate of potash in a dry year, as compared with the muriate, may be due in part to the difference in chemical activity of the calcium compounds formed where these materials are added, the sulphate being more insoluble and less active chemically than the chloride, and not having the power of replacing potassium, which is possessed by the latter under ordinary conditions.

On the whole, the hydrogen-ion concentration of the 1926 crop was more nearly neutral than the 1925 crop. This would tend to have a desirable effect on the burn in an indirect manner, as it would more nearly approximate the optimum conditions for oxidase activity in the curing and fermentation processes.

Summary and conclusions

This investigation had for its purpose the study of the effect of certain constituents of the leaf in relation to the burning quality of cigar-leaf tobacco. As a result of these studies the following conclusions seem justified:

1. The ether-soluble organic acids of the plant appear to occur almost wholly in combination with the alkali and alkali earth metals, as measured by the water-soluble alkalinity and insoluble alkalinity of the ash respectively.
2. In practically all cases there is a parallelism between the burning qualities and the water-soluble alkalinity of the ash.

3. The season and the form of potash supplied as a fertilizer affected the water-soluble alkalinity of the ash in practically all cases.
4. The phenomenon of base exchange in the soil and the removal of certain active substances through leaching probably have considerable to do with the burn and composition of tobacco, but the difficulty involved in the absorption of potassium, under different conditions, in highly colloidal soils such as those of the experimental plots, should also be given consideration.
5. The lime content of the samples used in this experiment was too high and the potash content too low for maximum burning quality. Further work is in progress relative to increasing the potash content and lowering the calcium content of the tobacco grown in Lancaster County, Pennsylvania.

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