

[54] **AIR REGULATION IN THE PYROLYSIS OF WOOD TO PRODUCE LIQUID SMOKE FOR THE TREATMENT OF FOOD PRODUCTS**

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[22] Filed: **Feb. 5, 1973**

[21] Appl. No.: **329,578**

[52] U.S. Cl. **426/221, 426/314, 426/332, 55/68, 55/73, 201/29**

[51] Int. Cl. **A23I 1/27**

[58] Field of Search 201/27, 34, 28-30; 426/312, 314, 315, 332; 202/182, 183, 184, 210; 55/68, 73

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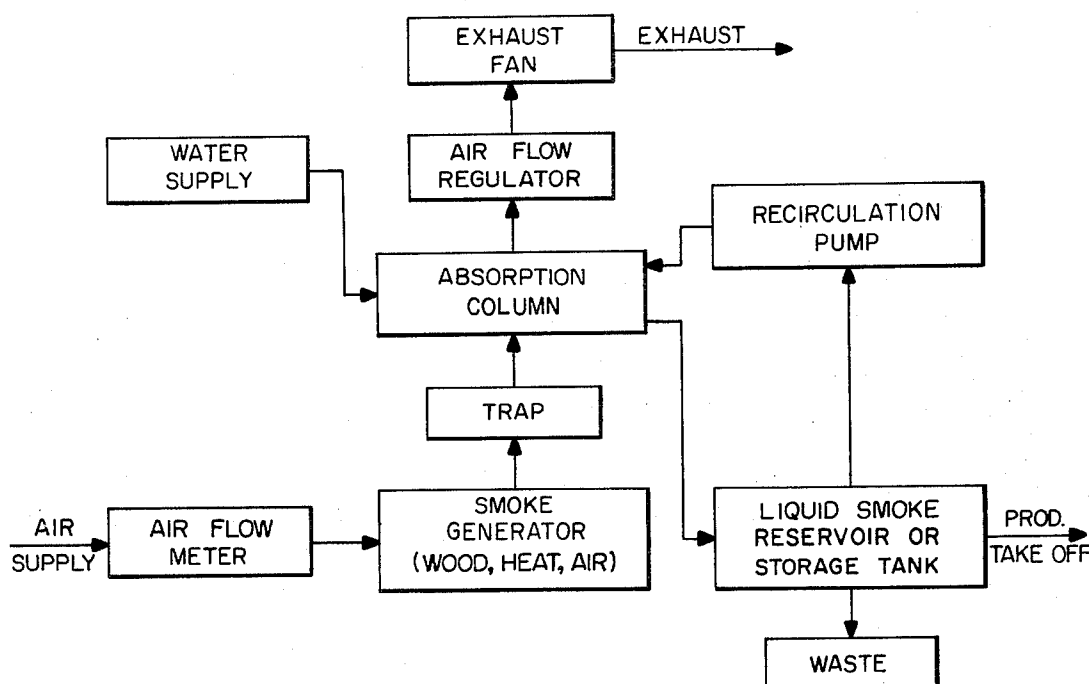
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[57]

ABSTRACT

A method for producing a liquid smoke product to be used in imparting a wood smoke stained appearance and a wood smoke taste to comestibles. The method involves the controlled carbonization combustion of wood in the presence of a regulated critical concentration of air, the air being introduced in an amount from about 3 to about 50 cubic feet of air for each pound of wood burned.

7 Claims, 3 Drawing Figures



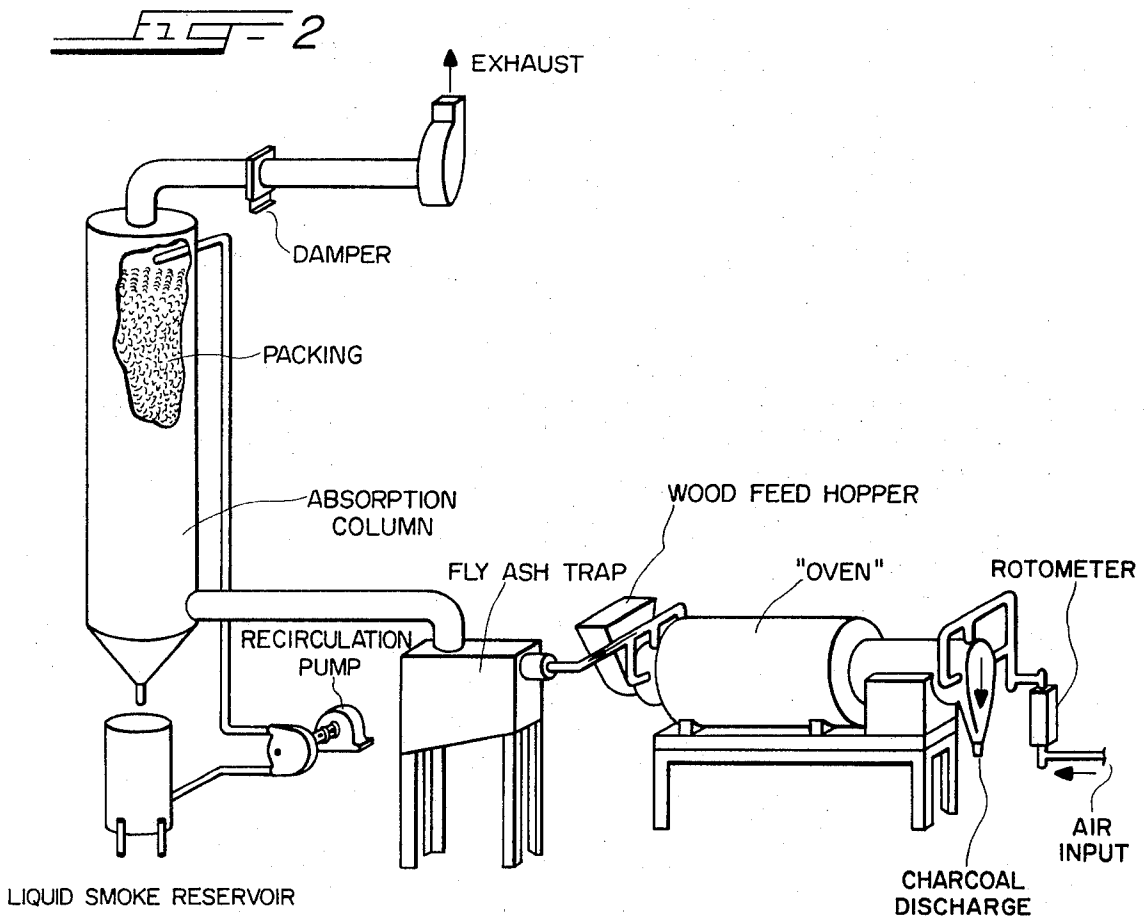
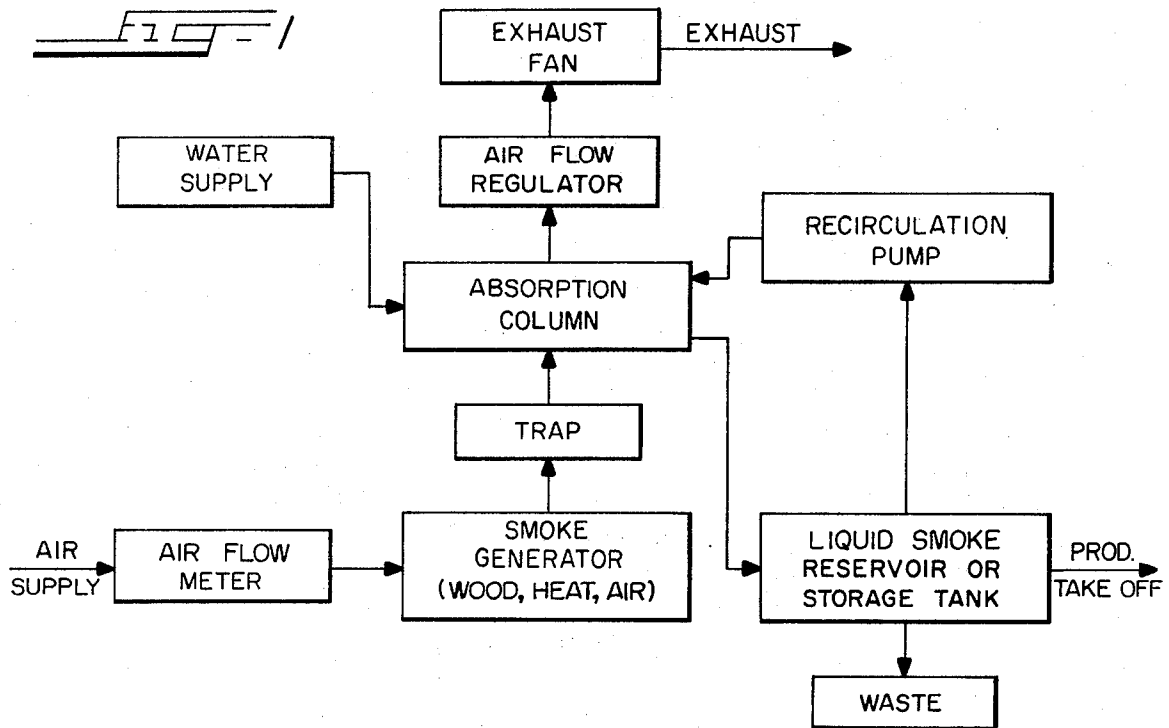
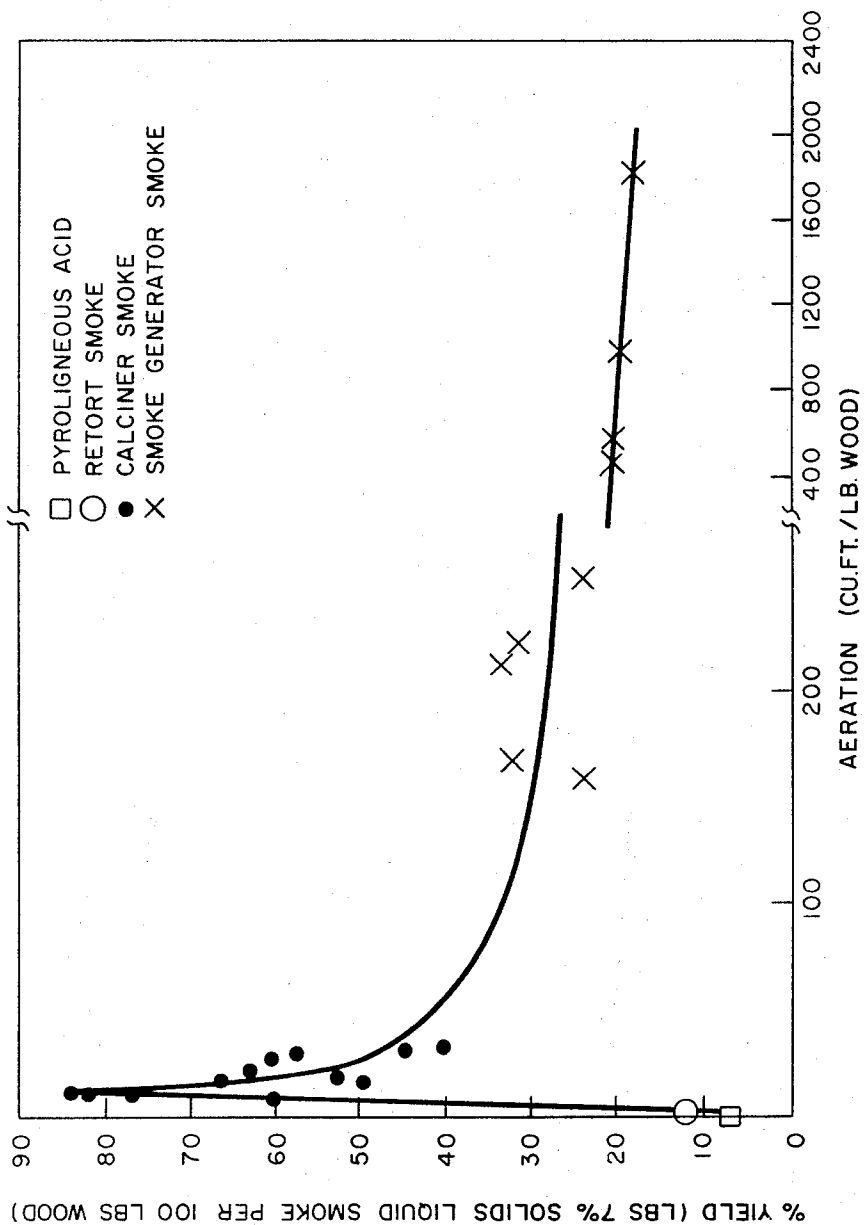


FIG - 3

EFFECT OF AERATION ON THE YIELD OF LIQUID SMOKE



AIR REGULATION IN THE PYROLYSIS OF WOOD TO PRODUCE LIQUID SMOKE FOR THE TREATMENT OF FOOD PRODUCTS

BACKGROUND OF THE INVENTION

The treatment of foods with wood smoke is one of the oldest means used in food preservation. In contemporary commercial practice the treatment with wood smoke is employed primarily for the characteristic color and flavor which are developed.

The prior art describes many different ways of exposing the food products to smoke or smoke media, and a widely adopted practice has been to expose the food products directly to a smoke atmosphere produced upon combustion of wood or sawdust. The food products, thus exposed to the smoke, would gradually take on the usual and desired smoky flavor and character, and the smoked appearance.

Smoke generators, or wood burning apparatus which has been used in the past to produce the desired smoke atmosphere, have certain inherent disadvantages the recognition of which has been a stimulus for extensive research for more convenient and more reliable and reproducible techniques. The smoke produced or obtained from wood fires cannot be effectively controlled as to concentration or density. Such a process cannot be relied upon to produce a product of consistent quality or character due not only to the variability in the woods used but also to the difficulty in controlling the burning process itself and, therefore, the nature of the smoke product produced.

Wood smoke is recognized to be a most complex chemical system including both vapor and particulate components. Except through the use of the most sophisticated equipment and regulatory apparatus, the smoke obtained from wood fires cannot be controlled either as to quality or quantity. As a result, there has been a recognized lack of uniformity of the smoked food product produced from plant to plant even when utilizing the "same" equipment in a direct smoking system.

Until the advent of more modern technology, the most common methods used in the direct smoking of food products have been the burning of damp hard wood sawdust in a batch operation, the heating of dry sawdust, or the burning of wood in a friction process. In any of these techniques the food product to be treated was suspended in a smoke house filled with the smoke or was subjected to the electrostatic precipitation of smoke particles onto the surface of the food product. In all of the techniques, the taste and the color imparted to the food is a function of the specific wood used and the technique employed, and differ from batch to batch.

In addition to the shortcomings of the techniques described above, the smoke house used in direct smoking has major shortcomings which have proven to be inefficient and to be undesirable for modern production goals. Original cost investments are objectionably high and frequent replacement of the mechanical components of the smoke generating equipment is required. There are continuous problems in maintaining the duct work adequately clean, and fires are not infrequent.

From the standpoint of ecology, the smoke houses have been notorious for creating air pollution problems. Deposits on the walls and duct work of the physi-

cal installation as well as in the fans and other equipment used are highly objectionable and necessitate time-consuming and costly cleanup in order to ensure adequate compliance with promulgated sanitation and safety standards.

The employment of conventional direct-smoking techniques has failed to solve the problems of guaranteeing uniformity in the smoky flavor or in the degree of color imparted to the food product, from batch to batch. The quality of treated comestibles has been most difficult to control, because the properties of the smoke itself have not been controllable. Additionally, the technique has been overly demanding in the time required to achieve the desired taste and color levels.

In the direct smoking methods particulate as well as vapor components of the smoke deposit on the food products being processed. Since it has been established that the particulate phase of the smoke often includes carcinogenic compounds, the deposit of such particulates on food products should be avoided.

Because of the many shortcomings and objectionable features inherent in the direct smoking methods, other techniques have been sought, and extensive research has been carried out in this area of food treatment. Among the alternate proposals has been the use of a product described as "liquid smoke," a substance which may be defined, generally, to include liquid reagents capable of imparting smoky hue or coloration and/or smoky flavor to a comestible exposed to the liquid or to its vapor phase. The use of liquid smoke has important advantages over the predecessor direct smoking techniques. For example, this preparation is much more convenient in use and has been considerably more effective in achieving reproducible smoke taste and smoke coloration in the products treated, provided adequate controls are utilized and appropriate precautions taken. Whereas the usual direct smoking technique ordinarily involves a batch process, it has been found feasible and convenient to use continuous processes when smoking with the aid of liquid smoke. The continuous processes have effected substantial cost savings, particularly since less elaborate equipment is needed than that called for in the prior art smoke house. Through the installation of relatively simple and reliable controls, the need for skilled personnel is also eliminated.

From a pharmacological viewpoint, it has been possible to eliminate from the liquid smoke the carcinogenic hydrocarbons and other compounds which are physiologically undesirable and which are normal constituents of wood smoke.

Several techniques have been found useful in adapting liquid smoke as a medium by which smoky flavor and a smoked appearance may be imparted to meet products and other comestibles. One procedure is to dip the food product directly into the liquid smoke. An alternative procedure is to treat the food products with a vaporized phase or with fine spray of the liquid smoke. In still other techniques the liquid smoke has been subjected to a flash evaporation step whereby the liquid product is nebulized or vaporized to establish an atmosphere effective in the "smoking" of the product contained in the treatment zone. Alternatively, a controlled quantity of the liquid smoke may be incorporated directly into the food product.

SUMMARY OF THE INVENTION

The present invention relates to an improved method and apparatus for producing liquid smoke, and to the products produced thereby. More particularly, the invention is directed to a process in which wood or wood products and equivalent raw materials are subjected to a controlled carbonizing combustion in the presence of a regulated air supply and an aqueous extraction system, whereby there is produced a liquid smoke having markedly improved flavor and significantly enhanced capabilities for coloring the food products treated therewith. Related important features of the improved liquid smoke product of the invention are that the liquid is readily dilutable, is produced in high yield, and is completely free of carcinogenic materials.

It is an important practical and commercial feature of the invention that the method is characterized by a high degree of reproducibility so that the resulting liquid smoke products are uniform in both their qualitative and their functional character, and in their effectiveness in use.

It is an important discovery of the present invention that there exists a critical relationship between the aeration level or the amount of air used during the combustion of the wood to produce liquid smoke, and those properties of the liquid smoke which are significant from a commercial standpoint.

While some general studies have been made in the past to investigate the nature of the chemical composition of wood smoke (Pettet and Lane, *A Study of the Chemical Composition of Wood Smoke*, *J. Soc. Chem. Ind.* 59, 114 (1940)), and while, generally, there exist data indicating an interrelationship between the nature of the chemical compounds contained in smoke preparations and the conditions under which the wood is burned, none of these data have shown the criticality of utilizing a specific, limited range of air concentration during the combustion process.

It is a related important feature of the present invention that, in addition to the effect of air in determining the precise chemical compounds contained in the smoke product itself, it has been established that the amount of air supplied during the combustion process is a most decisive factor in establishing the "quality" of smoke produced for use on food products.

Whereas data contained in the literature have reported an interdependence between the chemicals contained in the smoke combustion, these data provide little if any suggestion as to any criticality in the relationship as it may affect the quality of liquid smoke to be used in food processing. It is an important feature of the present invention that there has been established a critical airflow concentration which ensures optima in flavor, and dilutability of the liquid smoke produced.

It is a related important feature of the invention that within the discovered critical air concentration range it is possible to obtain a maximum yield of a high quality liquid smoke product.

Yet another important feature of the liquid smoke product produced in accordance with the method of the invention is that the liquid smoke is free of carcinogenic materials.

A related advantage of the method of the invention is that the liquid smoke produced is carcinogen-free without any need either for distillation or filtration, or cyclonic separation.

It is an important discovery of the present invention that the quantity of air needed to produce an optimum liquid smoke product is significantly less than the calculated air necessary to achieve "total combustion" of the wood itself.

An unpredictable and surprising discovery of the invention is that the amount of air to be used in producing an optimum liquid smoke product is about one-sixth of the calculated air required to effect complete combustion of the wood.

It has been established that in the pyrolysis of wood, increased aeration produces increased masses of glowing charred wood, promoting destructive distillation as well as establishing a highly oxidizing atmosphere, the two effects opposing one another. However, with increased air flow rates the contact time between the smoke product and the hot pyrolysis zone is shortened. This latter factor modifies the above-described effects.

It is an important feature of the invention that the process produces a remarkably uniform commercial product which is reliably duplicated from run to run and which has essentially constant food flavoring and food coloring capacities.

In a preferred embodiment of the invention water circulated through an extraction tower, and running countercurrent to the smoke produced, is effective to extract the smoke to provide the liquid smoke product of the invention.

It is a related feature of the invention that the water extract of the smoke evolved during combustion may be recirculated through a packed absorption tower further to concentrate the smoke elements in the extract solution, thereby to facilitate the production of a concentrate of any desired properties and strength.

DESCRIPTION OF THE DRAWING

FIG. 1 is a block diagram indicating the various functional components utilized in the apparatus for carrying out the method of the invention;

FIG. 2 is a perspective drawing illustrating, schematically, smoke-generating apparatus suitable for use in practicing the method of the invention; and

FIG. 3 is a graph illustrating the critical relationship between degree of aeration and yield of liquid smoke product.

DEVELOPMENTAL WORK AND ANALYTICAL TECHNIQUES

The prior art includes publications which refer to the use of air during the pyrolysis of wood. However, these references are singularly devoid of quantitative disclosures and are inadequate to provide sufficient information for those skilled in the art to calculate the precise aeration level utilized. For example, Hollenbeck U.S. Pat. No. 3,106,473 refers to the burning of wood "with a limited amount of air," and compares that procedure with the destructive distillation of wood carried out in the absence of added air.

Fessman U.S. Pat. No. 3,634,108 describes a wood smoke-generating process in which superheated steam is used in conjunction with air enriched with oxygen or a mixture of oxygen and inert gas. In each case the system is used directly in conjunction with the actual food smoking operation. However, the data presented are insufficient to permit calculation of any aeration levels achieved. Fessman claims as a preferred concentration the use of a mixture of oxygen and an inert gas in which

the oxygen is in the range of from about "0.5 to 5 parts by volume to 100 parts of steam by volume." It may be deduced that the oxygen level is extremely low since the ultimate product of pyrolysis is a "black carbon-like granulated residue, which can be burned again in air."

ANALYTICAL PROCEDURES FOR PRODUCT EVALUATION

In the experimental work carried out which culminated in the discovery constituting the method of the present invention, the liquid smoke product was analyzed with respect to several parameters including solids content, acidity, precipitable solids, flavor, and color factor. In each case the procedure adopted was the following:

1. Solids: an aliquot of the smoke solution was weighed on a predried standardized circle of Wattman No. 40 paper, and then dried for 2 hours in an oven at 105°C., in a forced draft.
2. Acidity: a 1.0 gram sample of the liquid smoke dissolved in 100 milliliters of water was titrated with standard NaOH solution to a pH of 8.15, and calculated as acetic acid.
3. Precipitable Solids: four milliliters of water was added to a 2.0 gram sample of liquid smoke and mixed, centrifuged 10 minutes at 2,400 rpm, and the supernatant discarded. The residue was washed with two 5-milliliter aliquots of water, dried 2 hours at 105°C. in a forced draft oven, and the weight of residue calculated as percentage of the initial sample.
4. Flavor: a triangle testing procedure using a nine-man panel was used. A sample of from 10 to 40 milligrams of liquid smoke was diluted to 250 milliliters in a soup containing 1 per cent salt and 4 per cent wheat flour. The panel was then asked to identify the odd sample on the basis of smoke flavor strength. In additional test procedures frankfurters previously dipped in a liquid smoke solution and processed in a smoke house were similarly evaluated.
5. Color Factor: color evaluations were conducted on both bacon and franks. In the bacon test 5 grams of the liquid smoke was sprayed on a 2 x 2 x 4 inch piece of cured pork belly which had been prewarmed by rotating for 8 minutes at a distance of 2 inches below a 250-watt infrared lamp. The bacon was then cooked by rotating an additional 15 minutes, after which it was chilled over night and the fat surface evaluated for smoked appearance. Frankfurters were dipped for 6 seconds in the liquid smoke, processes in a smoke house, chilled, peeled and evaluated for surface color.

The experimental investigation involved in development of the subject invention was carried out using three different types of smoke-generating equipment: laboratory and commercial retorts, a meat packing house type of smoke generator, and a rotating calciner. In the experimental work conducted, various types of hardwood were used. These included maple board, maple and hickory log sections, and maple and hickory sawdust. The equipment was controlled to achieve flameless combustion in each instance so that the ultimate solid product from the wood was charcoal rather than ashes.

LIQUID SMOKE PRODUCED IN CHARCOAL RETORT

An externally heated retort, provided with openings for the introduction of controlled quantities of air, was charged in each run with 1,000 grams of dry maple clippings. One run was conducted with no air added, and subsequent runs were carried out with aeration levels at 0.07 cubic foot of air per pound of wood and at 0.16 cubic foot of air per pound of wood. The results are tabulated below in Table 1.

Table 1

Effect of Aeration on Liquid Smoke Produced in a Laboratory Retort			
	Run No. 1	Run No. 2	Run No. 6
Aeration Level (cu.ft./lb.)	0.07	0.16	0
Carbonization Temperature (°F.)	437-887	437-734	437-734
Liquid Smoke Analysis:			
% Solids	8.4	9.4	10.8
% Acidity	15.3	15.3	15.4
Color Factor on Bacon	Slightly lighter than S-10	Darker than S-10	Lighter than S-10

In order to provide a meaningful reference and a useful basis of comparison between the liquid smoke product produced experimentally and the commercially available liquid smoke products, the experimentally produced products were compared with a commercial product herein identified as S-10. This reference product is advertised in the trade as having been produced in accordance with the teachings of Hollenbeck U.S. Pat. No. 3,106,473. That process involves the production of liquid smoke by burning wood in a "limited" amount of air, the smoke being absorbed in a recirculating countercurrent flow of water passing through a packed absorption column. A similar process is described in Ash U.S. Pat. No. 2,670,295. The Hollenbeck liquid smoke product is filtered through cellulose pulp to remove carcinogenic materials, while the liquid smoke product of Ash is distilled. Neither filtration nor distillation constitutes a process step in producing the liquid smoke product of the present invention.

A typical analysis of the commercial product identified as produced in accordance with the Hollenbeck process is as follows:

% Solids	8.7
% Acidity as acetic acid	9.9
% Precipitable solids	0.33

There are no data in the Hollenbeck patent indicating the aeration level employed or the carbonization temperature.

In a commercial carbonizing run, corresponding generally to the conditions set forth in Table 1, 40,000 pounds of wood (maple or hickory) per retort load was processed using 258 cubic feet of air for each 3,000 pounds of dry wood (0.086 cubic foot per pound), to produce liquid smoke at a yield of 11.7 percent by weight calculated on the basis of 7 percent solids in the liquid smoke product. This yield is a significant improvement over a 6.9 percent yield obtained in a pro-

cess in which essentially no outside air was supplied during the combustion cycle (the production of pyro-ligneous acid). The increase in yield is clearly attributa-ble to the addition of the air.

The liquid smoke product from several retort runs was combined and subjected to analysis, the results of which are set forth in Table 2.

Table 2

Characteristics of Smoke Product from Retort	
% Solids	10.6
% Acidity as acetic acid	18.8
% Precipitable solids	1.9
Flavor	2-3 times as "strong" as S-10, and somewhat harsh
Color factor on bacon	equal to or slightly darker than S-10

As indicated above, compared to the reference stan-dard S-10 the liquid smoke product from the retort is, as a practical matter, deficient in color since it was nec-essary to resort to a more concentrated product in order to achieve a comparable color factor. The prod-uct is strong in flavor but is inferior from the standpoint of dilution characteristics since it exhibited a high con-centration of precipitable solids (tar). The smoke prod-uct, after being allowed to settle several weeks, pro-duced a supernatant which was completely free of car-cinogenic materials.

In summarizing the work carried out with the retort apparatus, aeration levels up to about 0.16 cubic foot of air per pound of wood are effective to provide an in-creased yield over that obtained in the production of pyro-ligneous acid. However, the aeration level achieved is inadequate to develop the other desirable properties of a liquid smoke, that is, good flavor, good color factor, and good durability. Since increased aera-tion using the retort was not practical because of the hazard created by the presence of explosive mixtures within a totally enclosed structure, further experimen-tal work was carried out using a different type of appa-ratus, specifically, a packing house type of smoke gen-erator.

LIQUID SMOKE PRODUCED IN PACKING HOUSE TYPE OF SMOKE GENERATOR

In order to establish the effects of aeration levels higher than those obtainable utilizing a sealed retort, a series of investigations was carried out utilizing a pack-ing house type of smoke generator in which the carbon-ization of wood was conducted in a continuous, rela-tively open system. The generator itself constituted a 5-foot long horizontal tube or duct which was exter-nally heated and contained a coaxial, variable drive screw effective to carry and move sawdust at a rate of up to about 10 pounds per hour from a supply reservoir located at one end of the tube to a charcoal outlet at the opposite end.

The smoke generated in this machine was drawn up-wardly by means of a fan to pass through an absorption column packed with 1.7 cubic feet of 1-inch ceramic saddles, the smoke traveling countercurrent to a down-wardly flowing stream of water introduced at the top of the column from a reservoir therebeneath. This tech-nique and apparatus are similar to those described in Ash U.S. Pat. No. 2,670,295. The vapor effluent from

the column was vented through an adjustable damper system which served to regulate the draft of a fan as-sembly and, accordingly, the level of aeration occur-ring during the pyrolysis of the wood.

In accordance with the preferred embodiment of the method of the invention, the water was recycled to ef-fect a concentration of the desirable smoke compo-nents. The strength of the liquid smoke preparation was also controlled by regulation of the rate and the dura-tion of the wood pyrolysis and by adding water to re-place that lost through evaporation.

The packing house smoke generator made feasible the use of aeration levels as much as 1,000 to 10,000 times that possible with the retort. While normally such levels would tend to produce open combustion and complete ashing of the wood, by keeping the contact time of the air and the wood, and the external heat ap-plied at minima, it was possible to obtain flameless py-rolysis with charcoal as the end product. Yields of liq-uid smoke as a function of the aeration level are tabu-lated in Table 3, for air used at the rate of from about 160 to about 1,800 cubic feet per pound of wood. In each experimental run carried out 10 to 15 pounds of sawdust were used.

Table 3

Effect of Aeration Level on the Yield of Liquid Smoke Utilizing a Packing House Smoke Generator		
Experiment No.	Air Usage (cu.ft./lb. Wood)	Yield of Liquid Smoke at 7% Solids (% of Wood by Weight)
24-58	158	24
24-47	250	24
24-31	483	21
24-33	515	21
24-41	980	20
24-43	1820	18

The 18 to 24 percent yield of liquid smoke product, based on the weight of the wood pyrolyzed, is about twice that obtained in the sealed retort (11.7 percent yield). The data also show a decrease in yield with in-creasing aeration. This decrease may be due to loss of desirable smoke ingredients in the spent smoke dis-charged from the apparatus. Accordingly, it is impor-tant that the aeration rate not exceed the "capacity" of the equipment used.

The yield obtained from the smoke generator appa-ratus is also affected by the rate of recirculation of liquid in the absorption column. As indicated in Table 4, lower yields are achieved at relatively low recirculation rates.

Table 4

Effect of Recirculation Rate on Yield of Liquid Smoke Using a Packing House-Type Smoke Generator				
Expt. No.	Wood Pyrolysis Rate (lbs./hr.)	Aeration Level (cu.ft./lb.)	Recirculation Rate (ml/min.)	Yield of Liquid Smoke at 7% Solids (% of Wood by Wt.)
24-38	4.2	510	380-500	11
24-31	4.1	483	1800	21
24-33	4.3	515	3000-5000	21

When the size of the absorption bed (packed column) is limited, the rate at which the extracting water is recirculated has a compensatory effect, but only up to a point beyond which there is no further improvement. In an extension of the smoke generator work, reported below, the size of the absorption column was increased by a factor of about 10 (15.7 versus 1.7 cubic feet) and the absorptive surface of the column by a much larger factor through the use of ½ inch rather than 1-inch saddles. At the same time, the carbonization rate was increased by only a factor of 5. Under the conditions described, there were no apparent improvements in the yield when the recirculation rate was increased from about 3,500 to about 52,000 milliliters per minute. The results indicate that the absorptive surface of the column was more than adequate to accommodate the pyrolysis rate employed. The larger scale runs carried out with the smoke generator apparatus are summarized in Table 5 below.

The most significant practical effect apparent from the large-scale runs is that the yield of the smoke product was significantly higher, being about 33 percent as opposed to 20–24 percent for the smaller scale runs. It is believed that this increased yield reflects a more efficient absorption of the smoke components by full strength liquid smoke as compared with water. It will be appreciated that in the smaller scale experimental

runs most of the time of the run is utilized in building up the strength of the starting absorption fluid (water), while in the longer duration runs this period is relatively shorter and a larger fraction of the extraction time utilizes a liquid extractant which already includes a relatively significant fraction of the water soluble smoke components.

The liquid smoke products of run 24–75 and of run 24–83 of Table 5 were combined, aged one week to permit the settling of suspended tars, and then retained as liquid smoke product lot 24–89. A similar settling procedure was carried out with the liquid smoke product of Experiment 24–92 of Table 5, and the final supernatant product identified as lot 24–97. Lots 24–89 and 24–97 were analyzed to provide the data set forth in Table 6 below in which the properties of the product are compared with the S-10 standard.

The several effects of high aeration readily apparent. Color factor is built up to such a value that as little as 7 percent of solids in the smoke product provides a coloring capacity which exceeds that of the S-10 standard which has about 8.7 percent of solids. Since a liquid smoke preparation having 7 percent solids is completely suitable as a commercial product, the 7 percent solids product is used as a basis for comparisons in calculating yields, in the work reported herein.

Table 5

Experiment	Production of Liquid Smoke Utilizing Packing House-Type Smoke Generator		
	24-75	24-83	24-92
Sawdust:			
Total Usage, lbs.	80	110	130
Rate, lbs./hr.	7.4	5.5	5.9
Temperatures (°F.):			
Smoke, 4 inches above Bed	290–330	287–330	290–340
Smoke, 18 inches above Bed	254–275	260–280	250–285
Smoke, entering Column	178–222	168–214	160–215
Liquor, exiting Column	101–113	81–101	87–108
Aeration, cu.ft./lb.:	167	222	211
Liquid Recirculation, ml/min.:	6000	6000	6000
Yields:			
Charcoal, % of Sawdust	34	39	33
Liquid Smoke, % Sawdust	32	32	33
Liquid Smoke, ml at 7% Solids	11,815	15,960	19,400

Table 6

Analysis of Laboratory Prototypes of Drenching Liquid Smoke			
	Lot 24-89	Lot 24-97	Reference S-10
% Solids	8.0	7.0	8.7
% Titratable Acidity as Acetic Acid	7.2	8.3	9.9
% Precipitable Solids (1 + 2 dilution with water)	0.11	0.10	0.33
Flavor—Soup Test Color Factor on Bacon	67% Darker than S-10	80% Darker than S-10	100% Standard

Other effects of extremely high level aeration include the following:

1. A reduction in flavor as compared with the reference S-10. This is believed to be attributable to volatilization or oxidation and removal of smoke components, particularly acid components and phenols, such products being known to impart an acrid or medicinal taste to liquid smoke. A tempering of the flavor of the final product in the respect indicated is believed to be a desirable aim, since the product thus produced may be conveniently used even when only a mild smoky taste is desired.
2. A reduction in precipitable solids. It is believed that this is due to an elimination of "cracking" or solubilization of tars which ordinarily occurs when little or no air is used. Again, this feature is believed to be desirable in a commercial liquid smoke product.
3. Freedom from carcinogenic hydrocarbons. A sample of the lot 24-89, after settling for one week and being decanted from the insoluble tars, was found to be completely free of carcinogenic hydrocarbons, e.g. benzpyrene. This important beneficial effect is believed to be due to the insolubilizing effect of the air on tars, which then permits ready removal of the tar-carried carcinogens as a water insoluble fraction.

Upon comparing the data developed in the experimental work carried out with the sealed retort with the data from the packing house smoke generator, it may seem that each desirable property of the liquid smoke product is enhanced by increasing the aeration level. Such improvement would appear to hold true for yields as well as for flavor and color. However, further investigative work, in an aeration region or zone intermediate the very low aeration of the retort and the exceedingly high aeration of the packing house smoke generator, provided results which were not only unpredictable, but which were completely unexpected. Specifically, an important element of the subject invention is the discovery that there exists a critical aeration level in which the liquid smoke product yield is markedly superior to that achieved either at extremely low aeration rates (below 3 cubic feet of air for each pound of wood burned) or high aeration rates (above about 50 cubic feet of air for each pound of wood burned). Moreover, it has been discovered that the higher yields obtainable in this relatively narrow intermediate aeration zone are achieved without sacrificing the other desirable properties of the liquid smoked product. Surprisingly, it has been discovered that by carrying out the controlled combustion of the wood at an aeration rate within the limit defined, one can achieve an optimum balance between all of the desirable smoke properties on one hand and the yield on the other.

LIQUID SMOKE GENERATION USING A COMMERCIAL CALCINER

A commercial calciner was used as the means of conducting a series of runs to explore the effect of aeration rates intermediate those obtainable through the use of the sealed retort and the packing house smoke generator. A style "D" calciner manufactured by the Bartlett-Snow Company proved to be eminently suited for this work. Like the packing house smoke generator, the calciner included an elongated tube (6 inches in diameter and 7 feet long) which was heated externally to carbon-

ize sawdust passing from a feeder at one end of the tube to a charcoal discharge outlet at the other. However, whereas the packing house smoke generator used a screw or auger as the means for propelling the sawdust through the combustion tube, in the commercial calciner the entire tube was rotated. The rate of feed of the sawdust into the tube, coupled with the specific inclination of the tube with respect to the horizontal, regulated the rate at which the sawdust passed through the calciner.

Drawn through the calciner by means of an exhaust fan, the flow rate of the air was controlled by an adjustable damper positioned ahead of the fan. An accurate record of the volume of air passing through the tube was provided by means of a calibrated rotameter. The air was metered as it entered the apparatus at the charcoal discharge port. The air entering the apparatus passed over the pyrolysis zone and carried with it the smoke generated in the calciner, the smoke being drawn past the sawdust input end of the apparatus and into the duct work connected to the absorption column, all as indicated schematically in FIG. 2. In the particular embodiment of the apparatus used, the absorption tower was 2 feet in diameter and packed with 1/2 inch ceramic saddles to a height of about 5 feet, representing a volume of 15.7 cubic foot of packing. Countercurrent circulation and recirculation of water through the packed column effected an absorption of the smoke, in a manner similar to that described with reference to the packing house smoke generator. The spent gases exited from the column past a damper and a fan and were, thereafter, exhausted to atmosphere. Absence of significant exhaust smoke indicated substantially complete smoke absorption in the column.

The principal parameter studied utilizing the commercial calciner was aeration, which was explored in the range of from about 8 to about 45 cubic foot of air per pound of wood processed. Other operating variables were maintained essentially constant from run to run, and were as follows:

Wood used	Hickory, maple (8-15% moisture)
Wood feed rate	23 - 36 lbs/hr.
Wood processed per run	105 - 300 lbs.
Calciner rotation rate	9 rpm (Maximum)
Calciner pitch	1 inch per linear foot
Dwell time of wood in calciner	6 - 7 minutes
Pyrolysis zone temperature	345 - 600° F.
Temperature of smoke leaving calciner	160 - 238° F.
Temperature of smoke entering absorption column	141 - 194° F.
Temperature of recirculating liquid smoke	78 - 127° F.
Recirculation rate	3,000 - 53,000 ml/min.

For each run the temperature was at the lowest level at the start and climbed to the highest point as equilibrium was reached, generally within 1 hour. Similarly, the recirculation rate of the liquid smoke was lowest at the start and increased gradually throughout the run, correlated with a build-up in the liquid volume, which provided an increasing head of pressure to pump and, hence, an increased pumping rate. Variation in pumping rate had no appreciable effect on the results obtained since, as earlier demonstrated, the liquid smoke yield was the same whether the pumping rate was deliberately held to its low initial value or allowed to climb to its highest rate.

The various effects of adopting the "intermediate" aeration level (about 100 times that in the retort and about one-tenth of that in the smoke generator) are described in the following paragraphs, the principal variable or parameter affected being the overall yield of the liquid smoke product as indicated in the data of Table 7, and as depicted schematically in the graph of FIG. 3. The aeration rate in the calciner was varied from between about 9 cubic feet of air per pound of wood processed to about 34 cubic feet of air per pound of wood processed. Maximum yields were obtained at about 10 cubic feet of air for each pound of wood processed.

As also clearly indicated in the data developed, at the aeration levels utilized with the calciner the yields of liquid smoke obtained were significantly greater than the yields obtained utilizing other equipment and methods. For example, the calciner yield of liquid smoke was about 12 times that achieved under conditions producing pyroligneous acid, about seven times that obtained utilizing the very limited aeration of the retort, and three times that obtained utilizing the excessive aeration of the packing house smoke generator. These marked differences are differences in kind rather than in degree.

Within the aeration range investigated utilizing the commercial calciner, highest yields were achieved at the lower aeration levels, possibly because a lower aeration rate permits more thorough heating and more complete carbonization of the wood. This is evidenced by a decreased yield of charcoal and lower residual vol-

atiles in the charcoal. At higher aeration levels it was necessary to restrict and to limit the heat applied in order to reduce the danger of open combustion and accompanying ashing of the wood.

In order to learn what effect the extent of pyrolysis or carbonization might have upon the quality of the liquid smoke produced, a series of runs was carried out at a fixed aeration rate but with different extents of heating to produce 3 different levels of pyrolysis, each correlated with different yields of liquid smoke and charcoal. The results obtained are set forth below in Table 8.

Based upon the results tabulated above, it is evident that, with essentially a fixed rate of aeration, the more thorough the carbonization of the wood, the higher the yield of liquid smoke product and the greater the strength of that product, for a given volume of circulated water. A comparison of the properties of the three liquid smoke products indicates that, with increase in carbonization, the solids content, acidity, flavor and color factor increase. However, precipitable solids undergo a disproportionate increase as the treated wood nears complete carbonization. This is undesirable and can be dealt with by diluting the liquid smoke with water to reduce its strength to the commercially desired 7 percent solids level. During the dilution step a portion of the precipitable solids is thrown from solution and discarded, to yield a final product comparable, in soluble tar content, to the S-10 commercial reference product.

Table 7

Effect of Aeration Level on the Yield of Liquid Smoke Using a Calciner							
	10	6	Run Number				
			13	9	14	15	8
Aeration Level, cu.ft./lb. Wood	33.4	34	19	17.5	9.1	9.1	10.7
% Yield of Liquid Smoke (7% Solids)	40.2	44.9	52.9	65.9	76.8	81.8	83.8
% Yield of Charcoal	55.1	52.3	48.0	32.4	24.5	26.4	31.0
% Residual Volatile in the Charcoal*	59.4	60.3	63.2	39.7	30.0	30.6	54.8

*Measured by heating the charcoal 7 minutes at 950° C. to drive off residual volatiles without burning the charcoal.

Table 8

Effect of Degree of Pyrolysis on the Yield and Quality of Liquid Smoke			
	14	Run Number	
		17	16
Processing Conditions and Products:			
Aeration Level, cu.ft./lb.	9.1	8.3	8.5
% Yield of Liquid Smoke (7% Solids)	76.8	60.3	39.3
% Yield of Charcoal	24.5	33.4	63.5
% Residual Volatiles in the Charcoal	30.0	47.1	74.1
Analysis of the Liquid Smoke:			
% Solids	8.6	6.9	5.1
% Acidity as Acetic Acid	8.3	7.6	5.7
% Precipitable Solids	0.56	0.15	0.02
Flavor (% of S-10)	133	100	80
Color Factor on Bacon	Much darker than S-10	Darker than S-10	Comparable to S-10

There being no deleterious or objectionable properties or effects experienced other than indicated above, it is concluded that complete wood carbonization is a desirable goal of the overall process, since it affords maximum yields of liquid smoke product.

The importance of using water as a reactant or extractant in the liquid smoke generating process was brought out by conducting a run in which no water was charged to the reservoir of the apparatus and in which the recirculation pump was left "off." In this work, the results of which are tabulated below, the generated smoke was condensed in a tortuous network of pathways or channels in the packed column and was recovered as a smoke condensate in the reservoir below. Processing parameters were held fixed, as shown, with an aeration level of about 45 cubic feet per pound of wood used, to provide a liquid smoke product yield of 65.5 percent and a charcoal yield of 35.2 percent, indicating good carbonization. The properties of the liquid smoked product itself are indicated in the analysis set forth in Table 9.

Table 9

Analysis of Liquid Smoke Made by Condensation	
% Solids	13.0
% Acidity as Acetic Acid	14.0
% Precipitable Solids	2.8
Flavor	500% of S-10
Color Factor on Bacon	Darker than S-10

It is evident that both solids and acidity are significantly increased as compared with the product in which water was used and recirculated in the packed column. Additionally, the precipitable solids were found to be at an exceedingly high level, almost 20 times that found in run No. 17 summarized in Table 8. The total solids were about twice as high, and the flavor level about five times as great. Finally, and very significantly, a sample of a product decanted and allowed to settle for 1 week and analyzed for carcinogens was found to contain about 27 parts per billion of benzpyrene.

The results obtained in the experiment described point out a heretofore unrecognized effect of the use of water in conjunction with the liquid smoke generating process and suggest important interactions between the liquid smoke and the water in the absorption column. Specifically, restriction of the aeration to the pyrolysis zone and elimination of the effective interaction between smoke condensate and the air introduced into the system results in the loss of many of the potential benefits of aeration. Exceedingly high (objectionable) flavor, poor dilutability of the liquid smoke product, and significant concentrations of carcinogens result. The investigation suggests that an important part of the effect of aeration takes place in the absorption column through interaction of the air with the recirculating liquid smoke.

In order better to compare the smoke condensate (without water circulation) with the water-absorbed liquid smoke product earlier described, the former was diluted with water to provide a strength comparable to the latter. After settling out the precipitated tars and other insoluble materials, the clear supernatant liquid

smoke layer gave the following analysis.

Table 10

Analysis of Diluted Smoke Condensate	
% Solids	6.9
% Acidity as Acetic Acid	7.9
% Precipitable Solids	0.1
Flavor	200% of S-10
Color Factor on Bacon	Slightly lighter than S-10

While dilution with water was effective to precipitate the bulk of the "soluble" tars, to provide a product of generally good dilutability, the high flavor level of the condensate contributed to a high residual level in the water-diluted product. Moreover, the color factor content of the latter was low, indicating deficient color factor production in the original condensate, probable due to the absence of air interaction as previously discussed.

The process of the invention is remarkably effective in eliminating carcinogenic substances from the final liquid smoke product. A composite sample made by pooling aliquots of the liquid smoke produced in runs 8, 9, 12, 14, and 15, after allowing the products to settle for at least 1 week, was examined as a product representative of the method of the invention. Relevant conditions of the representative runs were: aeration levels in the range of 9 - 21 cubic feet of air per pound of wood, charcoal yields of from about 24 - 32 percent (indicating good carbonization), and liquid smoke yield of from about 63 to about 84 percent. The supernatant liquid phase was analyzed for benzpyrene, and a second analysis was carried out on the settled or precipitated tar sample. The results obtained are significant and conclusive. Whereas the supernatant liquid product was found to contain 0 parts per billion of benzpyrene, the tar sample was found to contain 665 parts per billion. These data support the conclusion that with proper aeration and recirculation a carcinogen-free liquid smoke product can be readily prepared, any carcinogens originally present in the combustion product being effectively separated by the settling out of the insoluble tars or residues. The latter were found to amount to from about 7.4-13.6 percent by weight of wood carbonized.

A second composite sample of the calciner-produced liquid smoke product was prepared by combining the products of 12 individual calciner runs, allowing 1 week of settling, and then decanting the supernatant from the precipitated tar residues. The data of Table 11 show the analysis of this lot (No. 26-74) and permit comparison with the product from the packing house smoke generator (lot 24-86), Table 6, and with the commercial, reference product S-10.

While the calciner-generated liquid smoke product has some properties which are quite similar to that of the commercial product S-10, the former exhibits a significantly improved color factor. As compared with the product from the packing house smoke generator, the calcined smoke clearly shows the effect of decreased aeration, namely increased flavor and somewhat increased precipitable solids. A commercially significant difference is the improved yield achieved through the utilization of the lower aeration rate of the calciner.

Table 11

Comparison of Calciner-Produced Liquid Smoke to Other Smoke Products				
	Calciner Liquid Smoke Range of Individual Runs	Pooled Smoke Lot 26-74	Smoke Generator Liquid Smoke Lot 24-89	Reference S-10
% Solids	5.1 -8.6	8.2	7.9	8.6
% Acidity as HAc	5.7 -9.9	8.3	7.2	9.9
% Precipit. Solids	0.02-0.56	0.31	0.11	0.33
Flavor (Soup Test)	80-133%	100%	67%	100%
Color Factor, on Bacon and Franks	Comparable-much darker	Darker than S-10	Darker than S-10	Standard

The present invention establishes the existence of a critical range of aeration in which the desired properties and characteristics of commercial liquid smoke are materially enhanced. The present invention has made possible improved flavor, greater dilutability, improved color factor content, a complete absence of carcinogenic material, and increased yields. While the principal work was carried out using hickory and maple sawdust as the raw materials, tests carried out indicate that hardwoods such as oak, birch, beech, and others will serve equally well. It will be appreciated that a controlled oxygen atmosphere may be used in place of raw air.

The improved liquid smoke product of the invention represents a marked advance over not only pyrolygneous acid but over other prior art products as well, including commercial liquid smoke preparations made using unknown but obviously insufficient levels of aeration. Finally, the present invention has established a critical range of aeration which ensures the production of a commercial product markedly superior to that obtained either at lower aeration levels or at higher aeration levels.

It will be appreciated that when using different processing equipment there may be some variation in the precise aeration rate, etc. which will provide the maximum yield of improved liquid smoke product. However, in view of the clear teachings of the present disclosure, and recognizing the criticality in aeration rate as set forth herein, those skilled in the art will be able, through simple trial runs, to select the appropriate values for each controlling parameter involved. Such selection of the optimum aeration rate and other variables for any particular apparatus will be feasible without any demand upon or exercise of the inventive faculty.

What is claimed is:

1. In the method of controlled carbonizing combustion of wood to produce a liquid smoke product for use in imparting a wood-smoke-stained appearance and a wood-smoke taste to comestibles treated with said product, which method includes the steps of:

heating wood in a chamber in the presence of an oxygen-controlled atmosphere to effect thermal decomposition of the wood and to generate smoke, contacting said smoke with water to provide an aqueous extract thereof and to condense and to recover from said smoke a volatile distillate contained therein, and

separating from said condensed volatile distillate water-insoluble components entrained therein;

the improvement comprising providing, during heating of the wood in the chamber, air in a concentration of from about 3 to about 50 cubic feet of air for each pound of wood carbonized;

thereby to produce in high yield a clear, readily dilutable aqueous liquid smoke product free from carcinogenic compounds,

said liquid smoke product being further characterized by enhanced smoke taste, smoke aroma, and food coloring capacity.

2. The method of claim 1 wherein the separation from said condensed volatile distillate of water insoluble components entrained therein is carried out by the steps of:

retaining the water extract of said smoke in a storage vessel in a physically undisturbed state for a finite time period to permit substantially complete precipitation of said water insoluble components from said water extract to provide two distinct layers in said vessel, said layers comprising a clear supernatant overlying a water insoluble tar-like residue, removing said clear supernatant from contact with said tar-like residue, and retaining said clear supernatant as said liquid smoke product.

3. The method of claim 1 and further comprising the steps of recirculating said aqueous extract of said smoke in a zone of contact with said smoke to increase in said extract the concentration of said volatile distillate contained in said smoke, thereby more effectively and efficiently to extract said smoke and to provide a liquid smoke product exhibiting enhanced smoke flavor and smoke-color-imparting capabilities when applied to comestibles treated therewith.

4. The method as set forth in claim 3 wherein the rate of recirculation of the water extract is in the range of from about 10 to about 80 liters of extract per pound of wood carbonized;

whereby the yield of liquid smoke product is increased, flavor and color factors are improved, and carcinogens are eliminated.

5. The method of claim 1 wherein, during heating of said wood in said chamber, air is provided in a concentration of from about 5 to about 30 cubic feet of air for each pound of wood burned.

6. The method of claim 1 wherein, during heating of said wood in said chamber, air is provided in a concentration of from about 7 to about 20 cubic feet of air for each pound of wood burned.

7. A liquid smoke product produced in accordance with the method of claim 1 and characterized by improved color imparting capabilities, enhanced taste-imparting characteristics, freedom from carcinogenic material, and a high degree of dilutability.

* * * * *