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Mattina et al.

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[54] **METHOD OF MAKING RECONSTITUTED TOBACCO HAVING REDUCED NITRATES**

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[52] **U.S. Cl.** ..... **131/143,** 131/17 R

[51] **Int. Cl.** **A24b 03/14,** A24b 15/02

[58] **Field of Search** 131/143, 140, 144, 15, 131/17, 2

[56] **References Cited**

UNITED STATES PATENTS

3,616,801 11/1971 Hind 131/143

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Attorney, Agent, or Firm—Breitenfeld & Levine

[57] **ABSTRACT**

Natural tobacco is extracted with water to produce a soluble extract and an insoluble fibrous residue. The extract is passed over an ion retardation resin to remove potassium nitrate from the extract. The resin is a rigid styrene-divinylbenzene cross-linked polymer lattice having attached quaternary ammonium groups, interwoven with a flexible linear acrylic acid polymer. Thereafter, the denitrated extract is recombined with the fibrous tobacco residue.

4 Claims, No Drawings

METHOD OF MAKING RECONSTITUTED TOBACCO HAVING REDUCED NITRATES

This invention relates to reconstituted tobacco, and more particularly to a method of making reconstituted tobacco from which a substantial proportion of the nitrate has been removed.

Reconstituted tobacco as such is well known. One way of producing reconstituted tobacco is to extract the soluble ingredients of natural tobacco, which preferably has been macerated or comminuted in preparation for extraction. The extraction is performed by use of water, and generally withdraws from 30 percent to 55 percent by weight of the starting material. An aqueous slurry is then formed containing the fibers, an by usual papermaking techniques, the slurry with or without additives is transformed into a selfsustaining web. The tobacco extract, which may be concentrated to a liquor, is then introduced into the web. The application of the extracted tobacco material may be achieved in any appropriate manner, as by spraying, saturating, or otherwise.

According to the present invention, after the extraction step but before the tobacco extract is recombined with the fibrous web, the extract is contacted with a resin which selectively removes ionic components. The invention has particular value in connection with reconstituted tobacco made from appreciable proportions of burley tobacco midribs since these contain substantial amounts of potassium nitrate. However, the invention is not limited to use with any specific type of tobacco.

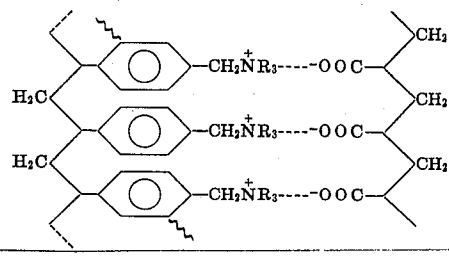
Removal of potassium nitrate from tobacco is desirable for several reasons. First, the burn rate of tobacco products is decelerated when the nitrate is eliminated. Secondly, the generation of several components in the smoke, among them oxides of nitrogen, methyl nitrate, and acetonitrile, is reducd. Some of these compounds have been suggested to be undesirable constituents in the smoke from the health standpoint. Furthermore, when potassium nitrate is burned, it produces an acrid smoke reminiscent of burned gunpowder.

In general, the idea of removing inorganic constituents from aqueous tobacco extracts is now new. In fact, removal of alkali metal salts from such extracts by passing the extract through an ion exchange resin is described in U.S. Pat. No. 3,616,801. However, the ion exchange resins referred to in that patent present certain problems. It has been found that although the resins described in the above-identified patent are effective in removing metallic ions from tobacco extract material, they remove almost all the desirable constituents of the extract as well. Furthermore, elution of the desirable tobacco components from the resins can be effected only with great difficulty. In addition, the anion exchange resins mentioned in that patent become fouled very rapidly and require cleaning steps to restore them to a usable condition after a single cycle of use.

All of these problems are overcome by the present invention. It has been found that by contacting aqueous tobacco extract with a particular resin, termed an ion retardation resin, the ionic material and specifically potassium nitrate, is selectively retarded while nonionic constituents of the extract pass through unaffected.

An example of the resin usable in this invention is manufactured by Dow Chemical Company of Midland,

Mich., and identified as AG 11A8. This resin is described in Dow Chemical Company Technical Bulletin "Ion Retardation" dated Nov. 1957. This type of resin is made by polymerizing an anionic (or cationic) monomer inside a "cage" of cationic (or anionic) exchange resin. Specifically, the retardation 11A8 resin bead consists of Dowex 1, a cross-linked styrene-divinylbenzene polymer lattice having attached quaternary animonium groups, interwoven with polymerized acrylic acid. The structure follows:



The quaternary ammonium groups are strongly basic anion exchange groups, and the carboxyl groups are weakly acidic cation exchange groups. Thus AG 11A8 absorbs both anions and cations in equivalent amounts. However, organic molecules are not absorbed by the resin. As a result, the original extract material which passes through the resin is desalted, while losing little or no essential tobacco constituents and after the denitrated extract is removed, the inorganic salts can be eluted from the resin simply by rinsing it with water. There is very little degradation of the valuable organic constituents of the tobacco extract during desalting with AG 11A8 since the resin is neutral, in contrast to the acidic and basic natures of conventional resins, such as those described in Pat. No. 3,616,801.

A typical procedure according to the invention involves extracting tobacco with water and then concentrating the extract so that it has a dry solids content of between one and 40 percent, and preferably about 10 percent. The concentrated extract is filtered or centifuged to remove suspended insoluble matter. Any suitable technique, such as those discussed in the aforementioned patent, may then be used to remove potassium nitrate using the resin described above. For example, the AG 11A8 resin may be arranged in a column and the aqueous tobacco extract passed through the column. Alternatively, a series of mixed contactors, in which the resin is stirred with the extract, could be employed. A continuous contactor, providing a moving bed of resin, as developed for continuous ion exchange, could also be employed. The denitrated extract may then be concentrated and used as is, or it may be mixed with other extract liquors to achieve a desired blend. Alternatively, natural tobacco may be blended in desired proportions before extraction and the extract obtained from the blend passed over the resin column to remove nitrates.

The following examples illustrate some of the features of the present invention:

EXAMPLE I

Burley tobacco midribs were extracted with water and the fibrous residue was formed into a paper-like sheet by ordinary papermaking techniques. The extract was concentrated, and passed through a column of AG 11A8 to effect removal of potassium nitrate from the extract. The denitrated extract was then applied to the sheet by means of a size press. As a control, reconstituted tobacco was made exactly as described above, except the extract was not passed through the resin column.

Both sheets were then shredded and made into cigarettes. The cigarettes were allowed to burn freely and the time it took for a 40 mm length of each cigarette to burn was noted. In addition, the smoke produced by the cigarettes was analyzed by gas chromotography techniques to determine the amount of nitric oxide and methyl nitrite in the smoke of each cigarette. The results of this testing are as follows:

Sample	Free Burn (minutes/40 mm)	Nitric Oxide (micrograms per cigarette)	Methyl Nitrite (micrograms per cigarette)
Control	3.0	800	650
Denitrated	9.5	100	30

EXAMPLE II

Denitrated extract liquor was prepared from burley tobacco midribs in the manner described in Example I. This liquor was blended with tobacco liquors prepared conventionally from flue-cured tobacco midrib and flue-cured tobacco dust to give a liquor with the following proportions:

30 percent burley midrib
30 percent flue-cured midrib
40 percent flue-cured dust

Reconstituted tobacco was made with this liquor and tobacco fibers in the same proportions set forth above. The material was shredded and fashioned into cigarettes. Control cigarettes were prepared from reconstituted tobacco of the same proportions but without removing the nitrate from the burley midrib liquor. The tests described in Example I were conducted with the following results.

Sample	Free Burn (minutes/40 mm)	Nitric Oxide (Micrograms per cigarette)	Methyl Nitrite (Micrograms per cigarette)
Control	6.2	300	215
Denitrated	7.2	65	20

EXAMPLE III

The procedure of Example 2 was followed except that the liquor had the following proportions:

50 percent burley midrib
10 percent flue-cured midrib
40 percent flud-cured dust The test results were as follows:

Sample	Free Burn (minutes/40 mm)	Nitric Oxide (Micrograms per cigarette)	Methyl Nitrite (micrograms per cigarette)
Control	5.5	650	360
Denitrated	6.6	85	25

EXAMPLE IV

A tobacco blend consisting of:
30 percent burley midrib
30 percent flue-cured midrib
40 percent flud-cured dust

was prepared and extracted with water. The extract was passed through an AG 11A8 resin column and concentrated. Reconstituted tobacco was made with this liquor and tobacco fibers in the same proportions as set forth above. The tobacco was shredded and fashioned into cigarettes. Control cigarettes were prepared from reconstituted tobacco made exactly as described above except that the liquor was not denitrated. The test results were as follows:

Sample	Free Burn (minutes/40 mm)	Nitric Oxide (micrograms per cigarette)	Methyl Nitrite (micrograms per cigarette)
Control	6.2	300	215
Denitrated	7.7	25	15

The test results of these examples show that cigarettes made with reconstituted tobacco containing extract material which has been denitrated according to this invention burn more slowly than otherwise identical cigarettes, and contain less of the compounds which otherwise occur due to the presence of potassium nitrate in the tobacco.

For flavor testing, cigarettes were prepared by blending shredded leaf tobacco with each of the four denitrated reconstituted tobaccos, and each of the four control reconstituted tobaccos, prepared in Examples I–IV above, the reconstituted tobaccos also having been shredded. In each case, the blend had a ratio of 70:30, leaf tobacco to reconstituted tobacco. The cigarettes were smoked by a panel of experts and evaluated

for flavor. In each case, the panel preferred the cigarette including the denitrated tobacco because the smoke was less acrid and more flavorful.

The invention has been shown and described in preferred form only, and by way of example, and many variations may be made in the invention which will still be comprised within its spirit. It is understood, therefore, that the invention is not limited to any specific form or embodiment except insofar as such limitations are included in the appended claims.

What is claimed is:

1. A method of making reconstituted tobacco, comprising the steps of:

a. extracting natural tobacco with water to produce a tobacco extract and a fibrous residue.

b. contacting the tobacco extract with an ion retardation resin to remove potassium nitrate from the extract, said resin comprising a cross-linked styrene-divinylbenezene polymer lattice having attached quaternary ammonium groups, interwoven with polymerized acrylic acid,

c. forming the fibrous residue into a paper-like sheet, and

d. recombining the denitrated extract with the fibrous tobacco sheet.

2. A method as defined in claim **1** wherein the natural tobacco includes burley tobacco midribs.

3. A method as defined in claim **1** wherein the extract which is contacted with the resin and thereby denitrated is obtained entirely from burley tobacco midribs, and the denitrated extract is blended with extracts of other types of tobacco prior to recombination with the tobacco sheet.

4. A method as defined in claim **1** wherein the natural tobacco which is extracted is a blend of different types of tobaccos, one of the types being burley tobacco midribs.

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