

## UNITED STATES PATENT OFFICE

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## DENICOTINIZING TOBACCO

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This invention relates to the preparation of smoking tobacco, more particularly to the treatment of the same to lower the nicotine content of the smoke. It is well known that during smoking the nicotine present in the tobacco volatilizes and tends to irritate the membranes of the mouth and throat of the smoker.

The present invention has for an object the treatment of cigarette, cigar and pipe tobacco to form compounds with the nicotine therein which will not as readily volatilize during smoking. For this purpose the leaf tobacco, shredded tobacco or scrap tobacco may be treated with certain substances which evidently form addition compounds with the nicotine to produce compounds which have a considerably lower vapor pressure and do not volatilize as readily during the smoking of the tobacco. For example, I have found that the compound formed by treating nicotine with  $\text{ZnCl}_2$  is very stable and will not evolve appreciable nicotine up to  $725^\circ\text{F}$ . which is  $284^\circ$  above its boiling point. Similar addition compounds are formed by treatment of nicotine with  $\text{MgCl}_2$  and  $\text{AlCl}_3$ .

Nicotine occurs in tobacco in both the free and combined state. In the combined form, it principally appears as a malic, oxalic and citric acid addition product. Under the conditions of temperature, etc., at which tobacco is smoked these addition products at least partially dissociate into the original nicotine which appears in part as such in the smoke. It is worthy of note that my addition compounds do not volatilize nicotine as readily as free nicotine. I have found that it is possible to form certain addition products which are more stable and which do not dissociate to nicotine as readily under smoking conditions. These more stable addition compounds allow the nicotine to burn, or polymerize and then burn, to such products as carbon dioxide, water and possibly nitrogen oxides.

Acids, such as hydrochloric, form addition compounds with nicotine.

Certain compounds, such as chromic, perchloric, picric and nitric acids form addition compounds with the nicotine in the tobacco whose oxidation is accelerated during smoking. For example, nitric, chromic and picric acids form addition compounds with the nicotine in the tobacco, and the heat of the cigarette flame during smoking causes the decomposition of the addition product into  $\text{CO}_2$ ,  $\text{H}_2\text{O}$  and other products. While with nitric acid there may be some nitration of the nicotine, there is also a partial addition compound reaction, which addition com-

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pound is oxidized during smoking. The use of any strong oxidizing acid is contemplated for the formation of compounds with the nicotine which are more readily oxidizable than nicotine. Moreover  $\text{Zn}(\text{NO}_3)_2$  and  $\text{ZnSO}_4$  form addition compounds with nicotine which are readily oxidized during smoking.  $\text{Zn}(\text{NO}_3)_2$  has oxidizing as well as addition properties. The burning of tobacco treated with  $\text{Zn}(\text{NO}_3)_2$  or  $\text{ZnSO}_4$  is not retarded, whereas the burning of tobacco treated with  $\text{ZnCl}_2$  is retarded. Accordingly, still another object is to form addition compounds with the nicotine which will oxidize more readily than nicotine during smoking.

In practically every case of treatment of the tobacco to form addition compounds with the nicotine there seems to be a reduction of tar content of the smoke. Evidently addition compounds are formed with the tar-forming compounds in the tobacco, such as those of pyridine bases, etc. Furthermore upon treatment of the tobacco with nitric acid, which forms addition compounds with the nicotine, and combines with the tar-forming nitrogen compounds, there is a considerable reduction in the tar content of the smoke. Likewise, treatment with perchloric, sulfuric, nitric, chromic and picric acids, which also form addition compounds with the nicotine and combine with the nitrogenous tar-forming compounds in the tobacco reduces the tar content of the smoke. Hydrogen peroxide also combines with nicotine to form nicotine peroxide. Other peroxides such as benzoyl peroxide, the ozonide of benzene and other unsaturated hydrocarbons react similarly with nicotine. Hydrogen peroxide also combines with the tar forming compounds of the tobacco to reduce the tar content of the smoke. Presumably the treatment of the tobacco with these and other oxidizing agents forms compounds with the nitrogen tar-forming constituents whose oxidation is accelerated during smoking. In the case of nitric acid, which was found to be a very good reagent for this purpose, the tar content in the smoke was lowered from 8% to less than 2%. A further object, therefore, is to reduce the tar content of the smoke from the tobacco by treating the same to alter the chemical constitution of the tar-forming materials so that the tar content of the smoke will be reduced. However it has been found that perchloric and chromic acids impart practically no odor to the tobacco smoke, whereas hydrogen peroxide and nitric acid when used in larger quantities impart a detectable taste in the smoke. Hydrogen peroxide and the other peroxides

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enumerated combine with nicotine in the tobacco to form nicotine peroxide compounds which volatilize less readily than nicotine and will oxidize more rapidly than nicotine during smoking.

It is trivalent nitrogen in the pyridine nucleus of nicotine that is principally affected by these various addition agents. By way of example, if zinc chloride is added to nicotine or to tobacco which contains nicotine, the zinc chloride adds directly to the trivalent nitrogen of the pyridine nucleus, forming a "pentavalent" nitrogen. If greater than molecular proportions of zinc chloride are added to nicotine then a second addition begins to take place on the pyrrole nitrogen.

The zinc chloride addition compound has been found to lower the nicotine content of the smoke by at least 40%.

As mentioned above, certain other compounds which are acid in nature also add to the nitrogen in nicotine to lower the vapor pressure of the nicotine. By the use of picric acid I have been able to lower the nicotine content of tobacco smoke from .48% to .17%.

In order to have a basis of comparison, a standard shredded tobacco was rolled into cigarettes. Five cigarettes were weighed and smoked on a puffing apparatus which was provided with proper absorption flasks. The nicotine was absorbed in dilute hydrochloric acid and this solution was then used to determine the percent nicotine. The silico-tungstic acid method was employed. This tobacco was used through Examples 1-5 inclusive, set forth below. It gave a value of 0.61% nicotine in the smoke, based upon the weight of tobacco burned during the puffing. The nicotine in the smoke in Examples 6 and 7 was also absorbed in hydrochloric acid and determined by the silico-tungstic acid method.

The following examples of treatment of the same standard shredded tobacco with the results of nicotine determinations are given in order to illustrate the invention:

#### Example 1

100 g. of shredded tobacco was sprayed with a solution of 4.02 g. of zinc chloride dissolved in 50 cc. of water. The percent nicotine in the smoke was found to be 0.23%.

#### Example 2

100 g. of shredded tobacco was sprayed with 120 cc. of a 5% solution of magnesium chloride hexahydrate. After the tobacco was allowed to dry the percent nicotine in the smoke was found to be 0.31%.

#### Example 3

20 g. of shredded tobacco was sprayed with a solution consisting of 3 g. of picric acid in 80 cc. of ethyl alcohol. The tobacco was dried on a steam radiator for 24 hours and the nicotine content of the smoke was then found to be 0.17%.

#### Example 4

20 g. of the shredded tobacco was sprayed with 20 cc. of a dilute nitric acid solution. The nitric acid solution was made up by adding one part of concentrated nitric acid (sp. gr. 1.42) to seven parts of water. The tobacco was then allowed to dry and the percent nicotine in the smoke was determined. The percent nicotine in the smoke was found to be 0.11% as compared with the original 0.61%.

#### Example 5

150 g. of shredded tobacco was sprayed with

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cc. of a 15% solution of hydrogen peroxide. The tobacco was oven dried, 2 hours, 100-110° C., and the nicotine content of its smoke was 0.35%.

#### Example 6

20 g. of a popular brand of cigarette tobacco which analysed to be 0.43% nicotine in the smoke, was treated with a solution of zinc nitrate to give a 7% zinc nitrate hexahydrate content by weight of the tobacco. After the tobacco was dried, the nicotine content of the smoke was found to be lowered to 0.20%.

#### Example 7

20 g. of a popular brand of cigarette tobacco was sprayed with 10 cc. of a 1-9 hydrochloric acid solution. The hydrochloric acid used was a 12 normal hydrochloric acid and was diluted with 9 parts of water. The resulting nicotine content of the smoke was found to be lowered from the original 0.43% to 0.30%.

What is claimed is:

1. The process of preparing smoking tobacco which comprises subjecting the tobacco to the action of a reagent selected from the group comprising perchloric, chromic and picric acids, said reagent reacting with the nicotine in the tobacco and forming therewith an addition compound which is less volatile than nicotine and will be oxidized during the smoking of the tobacco, and said reagent also combining with the nitrogenous tar-forming substances in the tobacco to form compounds which will be oxidized during smoking of the tobacco, and leaving the nicotine addition compound so formed in the tobacco to be oxidized during smoking.

2. The process of preparing smoking tobacco which comprises treating the tobacco with picric acid to form with the nicotine in the tobacco an addition product which is less volatile than nicotine and will be oxidized during the smoking of the tobacco, and leaving the addition product so formed in the tobacco to be oxidized during smoking.

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