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Thermal investigation of uncased and cased tobacco

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Abstract

Nicotiana tabacum, commonly called tobacco, has a huge economic importance worldwide. Among its varieties, Virginia has been chosen in this study. Its average chemical composition includes nicotine (2%), reducing sugars (22%), pectin (6%), fibers (7%), CaO (2%) and K₂O (2%), nitrogen (2%).

Thermal analysis showed to be an important tool to understand the process of leaf burning. TG/DTG and DTA were the methods applied using Perkin-Elmer and TA Instruments in inert and air atmospheres. Samples of uncased and cased tobacco were analyzed and the results showed loss of water and tobacco volatiles at around 80 °C. The main difference between cased and uncased samples lies in the start of oxidation. The production of CO and $CO₂$ began first in the cased unit, suggesting that added sugar may accelerate the reactions. The samples subjected to air atmosphere resulted in less residue, indicating that oxygen interferes with the reactions.

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1. Introduction

Nicotiana tabacum is a plant species the cultivars of which are of high economic and social importance in the sense that hundreds of families rely on it for their source of income. It is known as tobacco and one of the few nonedible cultivated plants and its trading is of the leaves.

Right after harvesting the leaves are cured which stage is the determinant of the type of raw material obtained. In Brazil, the cigarette industry has use chiefly of three types of tobacco which are flue-, sun-, and air cured. These are industrially known as Virginia, Oriental, and Burley, respectively [1]. Each

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tobacco type has own and important characteristics in the composition of a blend. By comparing the three types of tobacco after curing, one might generally say that Virginia is rich with reducing sugars and tend to medium contents of alkaloids and its combustion delivers aromas with green notes. Burley tends to be higher in alkaloids and nearly void of sugars, its prevailing notes being earthy and cigar aromas. Oriental tobacco contents of nicotine are low, levels of sugars are medium and aromatic notes remind of must. The average chemical composition of the tobacco types may vary considerably with the stalk position, climatic and soil conditions, cultural practices, types of curing, etc. Thermal analysis is an analytical process that allows to measure changes in chemical or physical properties of a substance or material as a result of the temperature or time under a controlled temperature program [2,3]. This study used three

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techniques of thermal analysis, the thermogravimetry (TG), derivative thermogravimetry (DTG) and differential thermal analysis (DTA).

2. Experimental

On the grounds that the burning tobacco reaches temperatures between 300 and 950 $^{\circ}$ C [4], the thermal analysis can be considered a most valuable tool for mapping chemical and physical characteristics of tobacco. Using this tool enables to measure kinetic parameters, in addition to investigating the reactions occurring during combustion of the material. In the specific case, the objective is to draw a comparative line between uncased and cased tobacco.

The tobacco type chosen for this analysis was Virginia, the average chemistry of which includes nicotine (2%), reducing sugars (22%), pectin (6%), fibers (8%), CaO (2%), K₂O (2%) and nitrogen (2%) [1]. The uncased tobacco sample was made at a stage immediately prior to the application of casing which consists basically of sugars, cocoa, licorice and humectant.

This trial used Perkin-Elmer Delta Series TGA7, 10° C/min rate of heating was used, with temperature varying from 50 to 900 \degree C in inert atmosphere. TA Instruments SDT 2960 equipment at a rate 100° C/min, with temperature varying from 25 to 800 \degree C in air atmosphere, respectively.

3. Results and discussion

For this analysis on a Perkin-Elmer with temperature varying from 50 to 900 \degree C (the maximum obtained by the equipment) in inert atmosphere.

Fig. 1 is the TG curves for the uncased and cased samples of tobacco in inert atmosphere. By overlapping the TG curves obtained for the two samples, one observes that from this point on the cased tobacco curve keeps drawing below the uncased tobacco. This would indicate a heavier mass loss during the thermal degradation process, however, without interfering with the raw material behavior in its original state. Comparing the mass loss of the two samples at 80 \degree C shows a mass loss around 6.0% for the uncased and 8.0% for the cased tobacco. This difference is not

Fig. 1. TG for uncased and cased tobacco, inert atmosphere.

considered significant. At the end of the run, the residual balance was around 5% for cased tobacco and 25% for cased tobacco. This would suggest that casing application interferes with tobacco reactions in inert atmosphere, resulting in a stronger oxidation.

Fig. 2 is the DTG curve for both samples. The cased sample built a slightly less pronounced declination curve from a temperature close to 80 \degree C, at which point the first peak appears on the DTG curve. This suggests the onset of loss of volatiles. The capital difference between the curves for the two samples was the second peak of the derivative curve at around 220 \degree C that appears more clearly on the cased tobacco curve. Maillard reactions and pyrazine formation as obtained from the reaction of sugars and tobacco components would be a possible explanation for this event. These reactions are desired as they impart a more pleasant aroma to the smoke. The onset temperatures found were

Fig. 2. DTG for uncased and cased tobacco, inert atmosphere.

238 °C for the uncased against 225 °C for the cased tobacco. This arouses suspicion that the cased tobacco reactions might have been accelerated with the addition of sugars which would in turn be reacting with natural tobacco components.

The largest amplitude peak appears on the derivative curves for the uncased and cased tobacco samples at about 350 \degree C, and in a more pronounced fashion for the uncased. The most abundant chemical group in tobacco is that of carbohydrates (pectin, cellulose, hemicellulose, starch, and reducing sugars) and this peak suggests oxidation of these components with CO and $CO₂$ formation. Because in this case the atmosphere was inert, the reaction might have taken place just with the components found in tobacco structure. This derivative peak is apparently an overlapping of two peaks, considering that a protrusion is observed to the left part of the curve. Probably because $CO₂$ formation was preceded by CO. The end of TG curves on both charts shows a residue of approximately 25% for the uncased and cased tobacco in inert atmosphere.

The analysis performed on a TA Instruments equipment provides some additional data to the trial in respect to the endothermic and exothermic peaks and the change in the atmosphere where combustion occurs. Also in this case, the heating rate was 10° C/min and the maximum temperature was 1000° C, although stability having been reached before 800° C.

Three charts were plotted from the two runs on cased and uncased samples of tobacco. Figs. 3 and 4 for TG and DTG, respectively. A comparison between

Fig. 3. TG for uncased and cased tobacco, compressed air atmosphere.

Fig. 4. DTG for uncased and cased tobacco, compressed air atmosphere.

uncased and cased tobacco curves in Figs. 3 and 4 shows the first peak to lie between 50 and 100 \degree C, thus suggesting loss of volatiles. Degradation of the cased sample was more remarkable up to around 430° C, which behavior is coherent with the analysis run on the Perkin-Elmer equipment. The onset temperature obtained for the uncased sample was $247 \degree C$ and the cased sample 147 \degree C, which signals that the reactions within the cased sample started at lower temperatures than in the uncased tobacco sample. These reactions may have been generated in the very components of the casing on the tobacco surface or in these components combined with tobacco constituents.

On the derivative curves for uncased and cased tobacco a second peak appears which would indicate the existence of chemical reactions that precede carbohydrate oxidation. The third and most pronounced peak occurs close to 430 \degree C, probably indicating degradation of plant cell wall carbohydrates (chiefly CO and $CO₂$). The mass residue is of around 7% for both samples, with the cased tobacco presenting a residual mass slightly higher than its uncased counterpart.

DTA thermograms in Fig. 5 temperature changes are observed in the samples which are due to endothermic or exothermic enthalpic transitions of the tobacco samples which vary in relation to a reference material. Both have similar behavior, with two exothermic peaks. The first peak is at a temperature close to 310 °C and the second one—more pronounced around 470 \degree C for the two samples.

Of the many possibilities of exothermic reactions, two are the most logical. The first possibility would be

Fig. 5. DTA for uncased and cased tobacco, compressed air atmosphere.

carbohydrate oxidation for both peaks from which one might infer that in the first peak CO formation occurs and in the second one, $CO₂$ is formed. The second hypothesis would be monosaccharide (first peak) preceding polysaccharide (second peak) oxidation The oxidation reaction appears to be slightly more intense for the uncased tobacco which would explain the higher evolution of heat.

4. Conclusion

Thermal analysis can be considered as a most valuable tool for the study of chemical reactions during tobacco combustion at temperatures close to real conditions to which tobacco is subjected when consumed. The most relevant features of this study are in the following items:

Cased sample mass loss was more pronounced at the start of the run in inert and compressed air atmospheres, possibly due to reactions of casing components on the leaf surface or the interaction of sugar molecules with natural tobacco components.

A slightly higher mass loss was found for cased tobacco in inert and compressed air atmospheres which is suggestive of the existence of chemical reactions that precede oxidation of carbohydrates as a result of the interaction between casing components or with the plant structure.

The third peak on the TG curves was the most pronounced for all the charts and might be correlated with the oxidation of carbohydrates the most abundant tobacco constituent with CO and $CO₂$ formation.

The residual mass balance was approximately 25% in inert gas atmosphere for uncased tobacco, considerably higher than the 7% obtained in compressed air atmosphere, probably due to carbohydrate oxidation reactions which took place in a more complete fashion in the presence of oxygen.

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