

RECOVERY OF NICOTINE AND NON-VOLATILE ORGANIC ACIDS FROM TURKISH TOBACCO WASTE

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Introduction

Tobacco waste consist of unusable parts of tobacco, obtained during the physical treatment in the production of cigarettes. Nicotine, oxalic, malic and citric acids, which are relatively more valuable than the other constituents of tobacco, can be extracted from the tobacco waste, using different solvents. There are many studies in the literature related to recovery of nicotine and organic acids from tobacco, using solvents (Rouver 1982, Garcia 1985, Li 1987).

1. Experimental

The tobacco waste used in this study was provided by a cigarette industry plant in Istanbul. The sample was spherical and had an average particle size of 0.25 mm. It contained 10 % moisture. A standard spectrophotometric method was used for the determination of nicotine content, which was found to be 1.28 % by weight, on average (ISO, 1974).

The amount of organic acids in the raw tobacco waste, was determined on the basis of total acidity, which was calculated as malic acid (Shmuk, 1961). The result was 8.7 g. organic acids per 100 g. tobacco waste.

The experimental set-up used in the study is shown in Fig. 1

100 g. of raw tobacco waste are extracted by using 1 liter solvent. The duration of the extraction and solvents used are given in Table 1 (Yildirim, 1993)

At the end of the extraction, the solid-liquid mixture was separated from each other. Nicotine and organic acids contents of the solid phase determined by standard methods, in order to calculate the extraction efficiency. Nicotine at the liquid phase was removed by steam distillation and was extracted from this solution by using chloroform. Nicotine content of this organic phase was determined by gas chromatography, in order to calculate the amount of nicotine lost during the process. Nearly 3 % of nicotine was lost during the process.

A GC equipment with a flame ionization detector (FID) and SE 30 type column was used for chromatographic analysis. Injection temperature was 230°C. A

nicotine-water solution containing 0.1 % by weight nicotine was used as a reference solution. Chromatograms of the reference solution and the samples are shown in Fig. 2.

The bottom product of steam distillation was a mixture of Ca^{++} salts of non-volatile organic acids in water. Ca^{++} salts were precipitated by adding H_2SO_4 as CaSO_4 and diluted organic acids were obtained from this mixture. Then organic acids were transferred to the organic phase with actone. Pure organic acids were obtained from this phase by evaporation of acetone. On average, 5.8 g. mixed organic acids were obtained from a 100 g. of tobacco waste by this procedure.

Organic acids in the raw tobacco waste and extracted solid phase, were converted into their methyl esters, using methyl alcohol solution containing a small amount of BF_3 (methylization solution) and their contents were determined by a GC, equipped with a thermal conductivity detector (TCD) and a carboxovax column. Injection temperature was 130°C.

Chromatograms of the solution produced by the methylization of the extracted solid phase are shown in Fig. 3.

2. Results and Discussions

The remaining nicotine contents at the solid phase and calculated extraction efficiencies are given in Table 1. Efficiencies are calculated using the following equation.

$$\eta_{1i} = (A_1 - B_{1i}) / A_1 \quad (1)$$

where A_1 is the total nicotine content of raw tobacco waste ($A_1 = 1.28\%$) and B_{1i} is the remaining nicotine content in the solid phase after extraction and filtration, in the experiment number i .

Yields of nicotine extractions carried out with different solvents and extraction times given in Table 1. Data in this table show that the most suitable solvent for the nicotine extraction is the saturated $\text{Ca}(\text{H})_2$ solution. The extraction yields increased as the extraction time increases for all solvents.

In order to determine the amount of non-volatile acids from GC results, the peak areas of organic acids were compared, as follows.

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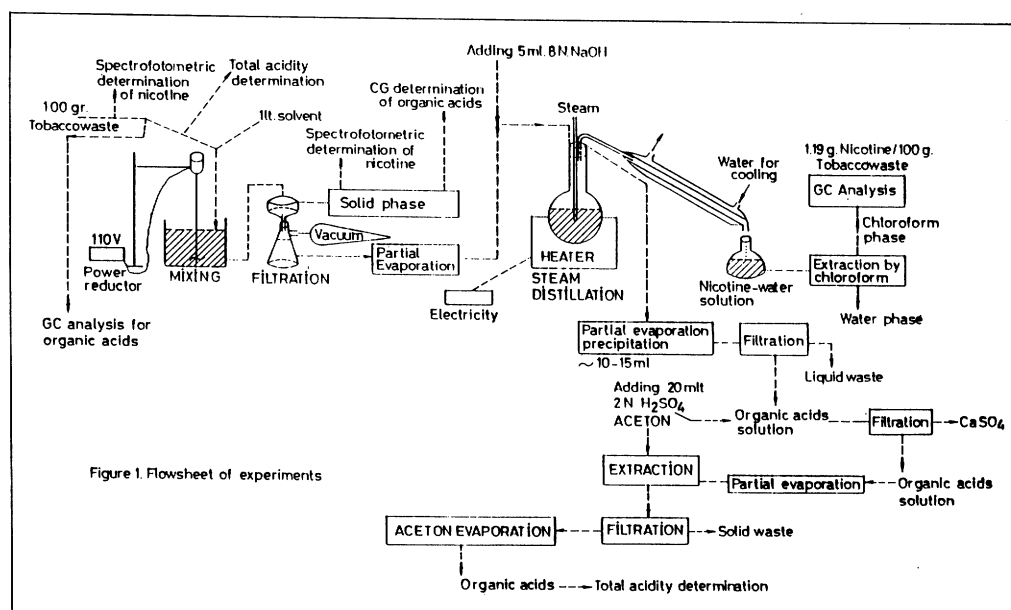


Fig. 1 Flowsheet of experiments

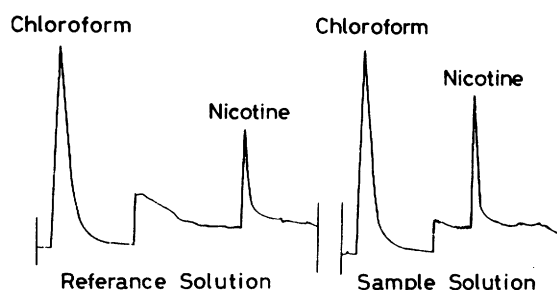


Fig. 2 Chromatograms for the determination of nicotine content in chloroform phase

Table 1 The amount of nicotine remaining in the solid phase and extraction efficiency

Extraction time [hr]	USED SOLVENTS					
	Saturated Ca(OH) ₂ solution + 10 % NaCl		Saturated Ca(OH) ₂ solution		Water	
	Nicotine (g.)	Efficiency (%)	Nicotine (g.)	Efficiency (%)	Nicotine (g.)	Efficiency (%)
0.5	0.35	72.6	0.30	76.5	0.33	74.2
1	0.30	76.5	0.22	82.2	0.25	80.5
2	0.24	81.2	0.19	85.1	0.22	82.3
3	0.18	85.9	0.13	89.8	0.17	86.7
4	0.14	89.1	0.11	91.4	0.14	89.1
5	0.10	92.2	0.05	96.1	0.09	93.7

$$\eta_2 = (A_2 - B_2) / A_2 \quad (2)$$

where A_2 is the area of the peak, obtained from raw tobacco waste ($A_2 = 49289$ for oxalic acid, $A_2 = 91967$ for malic acid), and B_2 is the area of the peak, obtained from GC analysis of extracted solid phase at each of experiments. B_2 values for two acids are shown in Table 2.

To determine the yields of organic acids, experiments were carried out using only extracts obtained with the saturated Ca(OH)₂ solution. It is clear from Table 2 that, the yields of organic acids extraction increases with the extraction time, like as nicotine yields.

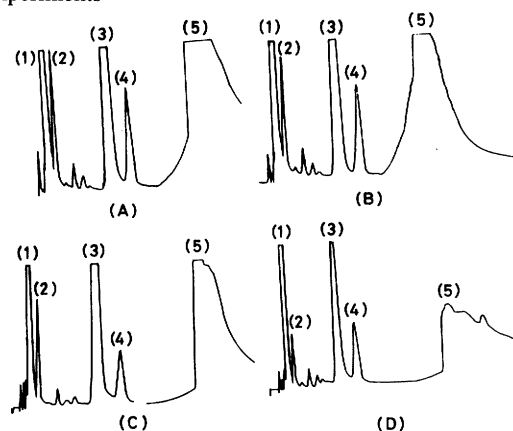


Fig. 3 Chromatograms of solution obtained from methylation of extracted solid phase

- (A): Solution obtained from raw tobacco waste, no extraction
 (B): Solution obtained by 1 hour extraction time
 (C): Solution obtained by 3 hour extraction time
 (D): Solution obtained by 5 hour extraction time
 Peaks: 1 Ethyl ether, 2 Oxalic acid, 3 Methyl alcohol, 4 Malic acid, 5 Water

Conclusions

The results of this experimental study can be summarized as follows:

- (1) It is possible to recover some of the nicotine from tobacco waste, with extraction using different solvents. The most suitable solvent is saturated Ca(OH)₂ solution. By using this solvent, efficiencies of nicotine extraction are high compared with others in a definite extraction time. Nicotine recovery increases with extraction time.
- (2) In the case of extraction with saturated Ca(OH)₂ solution, it is also possible to recover some organic acids, because Ca⁺⁺ ions are available in aqueous phase. Therefore saturated Ca(OH)₂ solution can be chosen for extraction of tobacco, because of simultaneous recovery of nicotine

Table 2 Results of GC analysis of samples, prepared from extracted solid materials by using saturated $\text{Ca}(\text{OH})_2$ solution

Components	Retention time [min].	Peak areas			
		Sample 1*	Sample 2*	Sample 3*	Sample 4*
Ethyl Ether	0.82	2656500	2558300	2266500	1667110
Oxalic acid	1.35	49289	32777	28667	14309
Methy Alcohol	3.98	619840	515790	52790	178530
Malic acid	5.04	94967	88971	72453	55810
Water	8.72	456820	964640	875325	171660
Oxalic acid yield, %	-	-	33.5	41.8	71.0
Malic acid yield, %	-	-	6.3	23.7	41.2

*

Sample 1 Solution obtained from raw tobacco waste, no extraction.

Sample 2 Solution obtained by 1 hour extraction time.

Sample 3 Solution obtained by 3 hour extraction time.

Sample 4 Solution obtained by 5 hour extraction time.

and nonvolatile organic acids.

(3) The results of GC analysis show that, about 71 % of

oxalic acid and 41 % of malic acid were recovered after an extraction time of 5 hours. But no citric acid was determined by GC analysis. Recovery of organic acids also increase with extraction time, as expected.

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