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# Characterization and level of desorption of volatile compounds released from boiling sweet potato pulp (*Ipomoea batatas*)

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## ABSTRACT

Sweet potato (*Ipomoea batatas*) is an important root crop in the Caribbean Basin. This crop releases volatile compounds that can be used to determine quality for acceptance or rejection of the food. The volatile compounds released by boiling the pulp of eight sweet potato varieties were identified, and the desorption process of some of them was studied. Volatile compounds were identified by static headspace extraction coupled with solid phase micro extraction (SPME), all of which allowed volatile analysis in a high water and carbon dioxide atmosphere. The eight sweet potato varieties studied were Ninety-nine, Pujols, Taiwan, Martina, Dominicana, Dune, Craneal and Miguela. Ten grams of sweet potato pulp paste (1:1 sweet potato-water) was sealed in a 20 ml headspace vial, and ethyl benzoate was added as internal standard (10 ng/g). The vial was heated to 80° C for one hour, and a polydimethyl siloxane (PDMS) solid phase micro extraction fiber was exposed to the sample headspace for one hour. The fiber was injected in a GC-MS. Some volatiles identified in all sweet potato varieties included hexanal, benzaldehyde, 2-pentilfurano, isoleudene, copaene and humulene. The Kovat indexes for DB-5MS analytical column were: 699, 846, 887, 1256, 1268 and 1342, respectively. Desorption isotherms of isoleudene, copaene and humulene showed an initial non favored process. Hexanal and benzaldehyde desorption were higher than those of simultaneous control samples between 10 and 200 ng/g. These findings suggest that the above mentioned terpenes were released from the matrix whereas aldehydes were synthesized during the boiling process. Results obtained from this study help to better characterize sweet potato and might be used to develop a quantitative selection index.

**Key words:** Sweet potato, SPME, Volatile compounds

## RESUMEN

Las batatas son un cultivo de importancia comercial en la región del Caribe. Este cultivo genera volátiles que se pueden usar para determinar la calidad de las variedades. En este estudio se caracterizaron los volátiles liberados durante la cocción en agua caliente de ocho variedades de batatas y se determinó el nivel de desorción de algunos de los compuestos identificados. La identificación de volátiles se hizo mediante la técnica de "static headspace" (SH) acoplada a microextracción en fase sólida

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(SPME). Esta técnica permitió el análisis de los volátiles en una atmósfera rica en humedad y contenido de dióxido de carbono. Las variedades estudiadas fueron: Ninety-nine, Pujols, Taiwan, Martina, Dominicana, Dune, Craneal y Miguela. Se utilizaron 10 g de una pasta compuesta de pulpa y agua en proporción 1:1 añadidos a un envase de 20 ml. La muestra fue enriquecida con etil benzoato como estándar interno, a un nivel de 10 ng/g. El envase fue sellado y calentado a 80° C por una hora, y luego una fibra acondicionada de polidimetil siloxano fue expuesta por 1 h adicional al HS de la muestra. Los compuestos adsorbidos en la fibra se analizaron por GC-MS. Se observó la presencia de hexanal, benzaldehído, 2-pentilfurano, isoledeno, copaeno y humeleno en todas las variedades de batatas. Los compuestos separados en una columna analítica DB-5MS de 30 m tuvieron índices de Kovats de: 699, 846, 887, 1258, 1268 y 1342, respectivamente. Las isotermas de desorción de los terpenos isoledeno, copaeno y humeleno adsorbidos en la pasta de batata presentaron un proceso de equilibrio inicial favorable. La desorción de los aldehídos hexanal y benzaldehído hacia la fase gaseosa fue superior a la de las muestras testigo en el intervalo de concentraciones de 10 a 200 ng/g. Se deduce que los terpenos son compuestos estructurales mientras que los aldehídos antes mencionados son generados durante el proceso de cocción de estas variedades. Estos resultados ayudan a caracterizar mejor estas variedades de batatas y pueden ser utilizados para obtener un índice de selección cuantitativo.

## INTRODUCTION

There are wide ranges in flavor within sweet potato germplasm (McLaurin and Kays, 1992), and many of these flavors depend upon a wide variety of aromas (Wang and Kays, 2000; Sun et al., 1995). These aromas appear during the cooking of sweet potato flesh. Starch is the major component of the flesh in sweet potato. Starch has been shown to form complexes with wide spectrum of molecules, such as aliphatic alcohols, ketones, aromatic aldehydes, cyclic alcohols and many others (Godshall and Solms, 1992). During sweet potato cooking, the formation of other carbohydrates besides starch, such as sugars, may determine the degree of volatile releasing (Lewthwaite et al., 1997). Mono- and disaccharides, such as glucose, fructose, sucrose and maltose exhibit a salting out effect on flavor compounds, causing an increase in their volatility in water because they are non-polar; thus they have low solubility in water. Some volatile compounds have direct impact on food flavor (Godshall, 1997) because they may increase or decrease taste intensity in food (Fennema, 1981; Wang and Kays, 2000). Therefore, to determine factors that govern sweet potato sensory qualities it is necessary to understand the mechanism involved in the retention, formation and releasing of flavors from the flesh.

The objectives of this study were to characterize volatile compounds present in eight sweet potato varieties and to study the sorption-desorption process for some volatile compounds identified. Those results are a part of a more ample effort to provide objective quantitative criteria for selection of sweet potato cultivars with good marketability characteristics that include sweetness, aroma and flavor.

## MATERIALS AND METHODS

**Field.** Eight sweet potato varieties were used in this study: Miguela, Martina, Dune, Taiwan, Dominicana, Craneal, Pujols and Ninety-nine. Varieties were planted in a randomized complete block design with four replicates at the Gurabo Substation, located in the eastern central part of the main island of Puerto Rico. Harvest was performed at approximately 150 days after planting. Roots weighing 400 to 500 g were selected at random from the field.

**Sample preparation and volatile compound analysis.** Sweet potatoes were cured by spreading the sample on a table for 7 days at 25° C. After curing process a sub-sample was cut to approximately 1 cm<sup>3</sup> and 300 g was poured on a blender with 300 g of water, and stored at -20° C until the analysis. An aliquot of 10 g was sealed in a headspace vial of 20 ml and it was cooked at 80° C for one hour. A polydimethylsiloxane (PDMS) solid phase micro extraction fiber was exposed for one hour to the headspace at 70° C. The fiber was then exposed to a solution of 200 µg/ml of ethyl benzoate in water for internal standard quantization.

The fiber was injected in a Perkin Elmer Gas Chromatograph Auto System coupled to a TurboMass mass spectra detector. The analytical column was DB-5MS 30 m X 0.25 µm id (Supelco Co.) by using 1 mL/min of helium as carrier gas.

**De-sorption isotherms of volatile compounds.** To determine de-sorption constants of sweet potato starch, 5 g of sweet potato flour was placed in a 50-ml screw-top gas tight bottle and brought to the moisture content of raw sweet potato (70%). Increasingly larger aliquots of the volatile compounds were introduced into a series of two parallel sets of vials, one containing a constant amount of the sweet potato starch, whereas the other contained 10 ml of water. Thus, there was always a pair with the same amount of the volatile compounds, so that the resulting difference in the area values corresponded to the adsorbed amount. The temperature in the tubes was brought to 70° C and a solid phase microextraction of PDMS was left exposed by 1 h. Then the fiber was exposed to a solution of 200 µg/ml of ethyl benzoate (internal standard) for 10 min. The SPME fiber was injected on a GC-MS.

## RESULTS AND DISCUSSION

The releasing of volatile compounds from the pulp of eight varieties of sweet potato was studied. Volatiles found in the headspace were determined by the use of the solid phase micro extraction (SPME) technique. Optimal temperature to determine volatile composition on headspace was determined by using six volatile compounds found in the headspace of the eight cooked sweet potatoes. These volatiles were benzaldehyde, hexanal, isolefene, copaene and humulene. Optimization of the temperature for extraction of the above mentioned volatiles by using PDMS-SPME showed that it was between 50 and 70 °C for most of the volatiles tested, so temperature choice was 65° C (Figure 1).

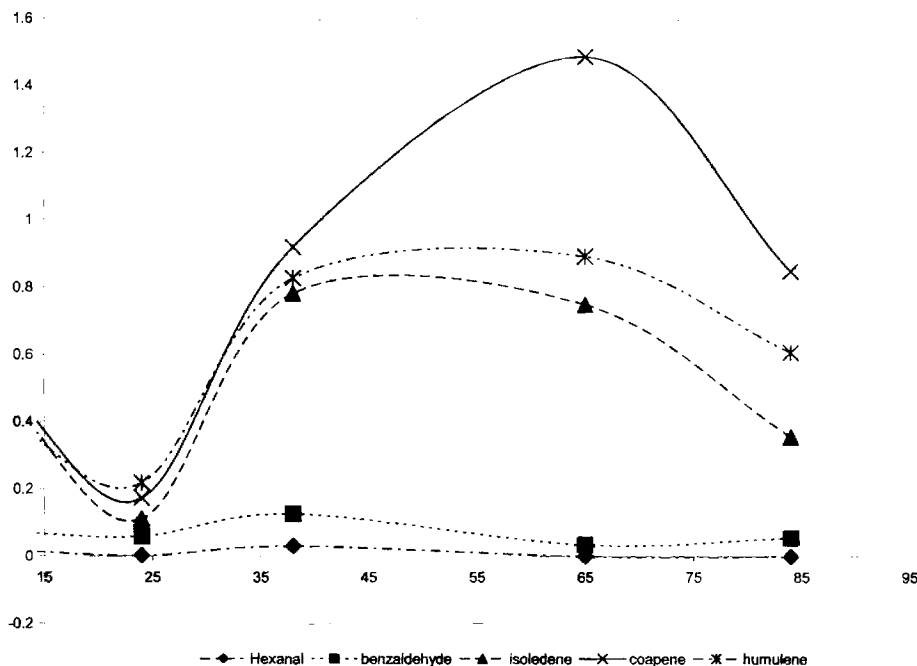


Figure 1. Profile of selected volatiles released from sweet potato pulp with temperature by using static headspace-SPME.

Volatile constituents of eight sweet potato types were isolated by static headspace sampling of the cooked pulp. Table 1 shows the amount of terpenes, oxygenated and non-identified compounds per sweet potato variety. Volatile compounds belonging to chemical families such as alcohols, ketones, aldehydes, aromatic hydrocarbons, esters and terpenes were found.

Table 1. Types of compounds found in the headspace of sweet potato

Sweet Potato Variety	Terpenes	Oxygenated Compounds	Non-Identified Compounds
Martina	16	12	10
Miguela	14	18	9
Dominicana	16	19	14
Taiwan	8	13	18
Dune	20	15	10
Pujols	16	10	12
Craneal	6	16	9
Ninetynine	19	10	9

Most of the odorants were identified on the basis of retention index and mass spectra and/or odorant standards. The chemical structures of some odorants identified suggest that they were present in raw pulp, but others suggest that they come from

Maillard-type reactions. Some of these odorants have fatty notes that at high concentration may come to be off-flavors. Other odorants have earthy-sweet notes that may come to be main contributors to good taste of sweet potatoes.

The same above mentioned six volatile compounds for optimization of the extraction method were chosen for preparation of desorption isotherms from sweet potato pulp. To measure the desorption isotherms we used a modified version of the VPC method. Increasingly larger aliquots of the selected volatile compounds were introduced into a series of two parallel sets of vials. One set contained a constant amount of the hydrated sweet potato pulp, whereas the other set contained a constant amount of water so that the volume of headspace on both sets of vials was the same; thus we did not need to make corrections for the sample volume. We used water as the parallel set because sweet potato pulp is at least 70% water. This experiment, as designed, gave volatile desorption measures. The differences between relative volatile pressure of sweet potato set and their corresponding water sets gave us the amount of volatile retained by the pulp (Table 2).

Table 2. Relative partial pressure of selected pesticide released from sweet potato pulp respect to control samples in water.

Hexanal Pp	Benzaldehyde		Humulene	
	(Pa-Pp)	Pp	(Pa-Pp)	Pp
0	0	0	0	0
0.005055	-0.004570	0	8.57157E-05	0.002475
0.008831	-0.007419	0.001678	-0.001274	0.036603
0.008979	-0.005971	0.002788	-0.001844	0.055009
0.016254	-0.009643	0.010002	-0.008170	0.147679
--	--	--	--	0.299608
--	--	--	--	0.376737

Copaene		Isoledene	
Pp	(Pa-Pp)	Pp	(Pa-Pp)
0	0	0	0
0.001209	-0.00020	0.002268	0.000486
0.015217	0.002795	0.040034	0.039512
0.021666	0.024828	0.060648	0.154626
0.049226	0.023664	0.170007	0.171978
0.116518	0.198597	0.35086	0.675223
0.137599	0.393314	0.423855	1.470424

Pp and Pa are area ratios of sweet potato and water sets, respectively.

The terpenes were found at very low concentration in raw pulp all of which suggests that they are released during cooking but not synthesized. Isoledene and humulene had sigmoidal desorption isotherms, all of which indicates enhanced volatile release from cooked pulp after a given pulp concentration (Figure 1).



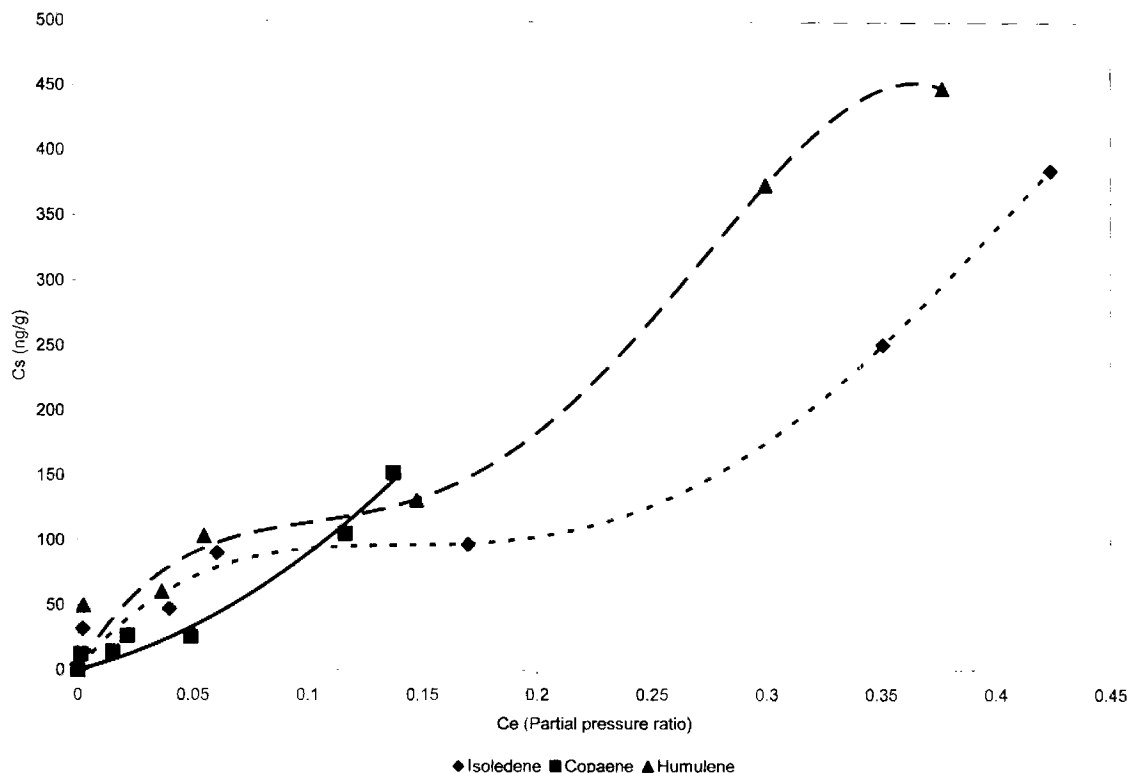


Figure 1. Desorption isotherms of Isoledene, Copaene and humulene determined by using static headspace-solid phase micro extraction technique.

However, the copaene isotherm had an upward behavior and its headspace partial pressure at a given pulp concentration was higher than that for isoledene or humulene (Table 2). These data suggest that copaene was released from sweet potato pulp more easily than isoledene and humulene.

Hexanal and benzaldehyde were not found in headspace of raw sweet potato pulp, but both were found in the headspace of all cook sweet potatoes. Desorption behavior of above mentioned aldehydes suggests that both of them were synthesized during cooking because the amount of each in sweet potato headspace was higher than in the parallel water matrix samples. Hexanal is a compound that has been associated with the Maillard reaction as well as other compounds found in the headspace of our samples. We found a negative desorption behavior for hexanal and benzaldehyde all of which indicates a high repulsion by cooked sweet potato pulp for both aldehydes (Table 2).

This study focused on the identification of volatiles and resulted in finding a number of compounds, some of which contribute to the overall aroma of sweet potatoes. Many of them were unique for one particular variety of sweet potato. Some off odors were identified which at high concentration could affect product acceptance by consumers. Some potent odorants such as benzofuran derivative, esters, aldehydes, and terpenes, were found at detectable concentrations by the use of static headspace coupled to SPME techniques, all of which showed the adequacy of this procedure for analysis of volatiles from cooked sweet potato pulp. Desorption data suggest that cooking sweet

potato pulp promotes the releasing of some volatiles, which may affect flavors when the processed product has been stored for long time.

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