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ORIGINAL ARTICLE

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## **Study on the retentivity of the volatile components of simulated guava juice using ultrafiltration**

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### **Abstract**

**Khalil, S.H., Aroua, M.K. and Sulaiman, N.M.**

**Study on the retentivity of the volatile components of simulated guava juice using ultrafiltration**

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An experimental investigation on retentivity of major guava juice volatile compounds during ultrafiltration is presented in this paper. Simulated guava juice solutions were used throughout the study and the ultrafiltration was carried out in a batch stirred cell system. Polyethersulfone membranes with MWCO of 100,000 and 500,000 were used. The effects of important process operating parameters such as pressure and pectin concentration were studied. It was found that hydrophobic interactions with the membrane are the major factors, which control the flavor retention. A qualitative correlation between component polarity and retentivity was also observed.

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**Key words :** volatile components, guava juice, ultrafiltration, retentivity

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Membrane processes are currently widely used for the clarification and concentration of fruit juice. Applications of ultrafiltration and microfiltration included clarification of apple, kiwi (Heatherbell *et al.*, 1977 and Wu *et al.*, 1990), star fruit (Sulaiman *et al.*, 1996) juices etc.

The presence of macromolecules such as pectin substances in the juice is detrimental to the performance of the membrane unit due to their fouling character. Most of the previous investigations focused on the effect of pectin on the permeate flux. No study is reported on their effect on the ultrafiltration behavior of flavor compounds, which are important constituents of any juice. Furthermore, studies on the ultrafiltration of guava juice are still scarce in the literature. This important subtropical fruit is known to contain high quantities of high-methoxyl pectic substances (Ferro *et al.*, 1969). Despite the use of pectinase enzymes during guava juice processing, a major part of the pectic substances remain in the final juice and can have a negative effect on the permeation of flavor compounds through the ultrafiltration membrane.

The main objective of this study is to investigate experimentally the behavior of major guava juice compounds during ultrafiltration in presence of pectin. Nine flavor compounds were selected for this study which used simulated solutions.

## Materials and Methods

### Materials

A total of eight major flavors were used in this study (Stevens *et al.*, 1970 and Yen *et al.*, 1990). The selected flavors were, 1,8-cineole, capronaldehyde, methyl benzoate, ethyl benzoate, 2-phenylethyl acetate and, cinnamaldehyde supplied by FLUKA, 1-hexanol and  $\beta$ -caryophellene from TCI. Ethyl cinnamate, which was used as internal standard supplied by MERCK, n-pentane and diethyl ether provided by BDH and FISCHER were used for flavor extraction. All these chemicals were of more than 98 % purity.

Pectin was from citrus fruits with galac-

turonic acid content of 79 % and methoxyl content of 8 %. The used pectin has a molecular weight ranging from 20,000 - 100,000 Dalton and was provided by SIGMA.

### Ultrafiltration membrane equipment

A 500 ml laboratory scale stirred cell unit was employed in this study, which has an effective filtration area of 38.5 cm<sup>2</sup> and fitted with polyethersulfone ultrafiltration membrane of 100,000 then 500,000 MWCO.

### Preparation of simulated juice

The simulated juice was prepared by adding 10  $\mu$ l of each of ten selected flavors using 10  $\mu$ l micro-syringe to 1 liter of pectin aqueous solutions with different concentrations of 0.01%, 0.025%, 0.05%, 0.075% and 0.1%.

### Ultrafiltration run

The ultrafiltration experiments were carried out at room temperature under applied pressures of 2, 5 and 7 bar. For each experiment the stirred cell would be filled up to 400 ml of simulated guava juice as a feed, and for every 40 ml of permeation from the stirred cell, it would be refilled again to the 400 ml level. A total of 180 ml of permeate was collected for flavor extraction.

### Flavor extraction

The flavor compounds were extracted using a mixture of n-pentane-diethyl ether (1:1) solvent. The extract was precisely concentrated to a final volume of 1 ml. The technique used here was developed and optimized in our department. The reproducibility of analysis is estimated to be within 5 %.

### Analytical method

GC-FID method was used to determine the concentration of the flavor compounds. Helium was used as a carrier gas at a flow rate of 1 ml/min. A split ratio of 1:1, and split flow rate of 1 ml/min was used. Oven program started at 40°C for 8 minutes and then increased at a rate of 4°C/min and ended at 200°C, remaining these for 20

minutes. 1.8  $\mu$ l of each ten flavor compounds with 1.8  $\mu$ l of internal standard (IS) were injected separately.

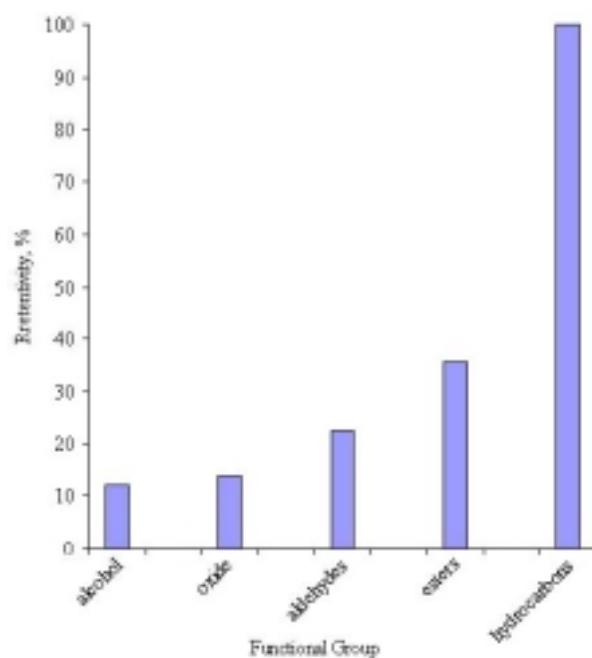
## Results and Discussion

### Effect of hydrophobicity

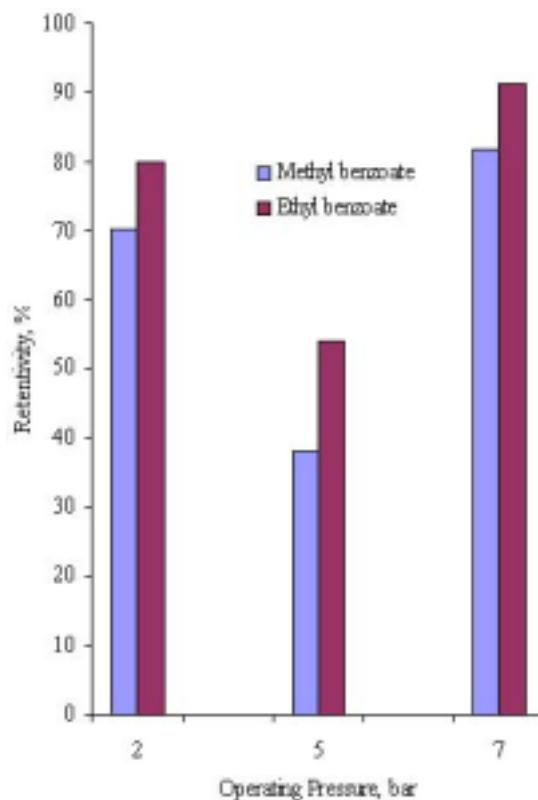
The results for the 100,000 MWCO membranes of the functional groups without pectin are illustrated in Figure 1. Significant rejections ranging from 12 % for 1-hexanol to 100 % for  $\beta$ -Caryophellene were obtained. These high retentivities are not expected since the molecular weights of these components are very much smaller than 100,000 Dalton. The observed retentivities were probably due to hydrophobic interactions with the membrane, which can result in adsorption of the components on the inner walls of the membrane pores and on surface of the membrane.

The ultrafiltration behavior of methyl benzoate and ethyl benzoate is an additional con-

firmation of the hydrophobic mechanism of their retention. These two components represent a homologous series (components with the same functional group and differing only in the number of carbons on the alkyl group). It is well established that in homologous series, a molecule with the greater number of carbons in the alkyl group is more hydrophobic than one with fewer carbons in the alkyl group. In all ultrafiltration runs with different operating pressures, it is found that ethyl benzoate is characterized by higher retentivity than methyl benzoate. This result, that is shown in Figure 2 for operating pressures of 2, 5 and 7 bars and 0.1% pectin concentration, is in concordance with the order of polarity of these two molecules.



**Figure 1.** Retentivities of the functional groups without pectin, using 100,000 MWCO membranes at 2 bar.



**Figure 2.** Effect of the number of carbons in the alkyl group of two homologous flavor components on their retentivity, for different operating pressures and 0.1% pectin concentration solution.

### Effect of pectin concentration

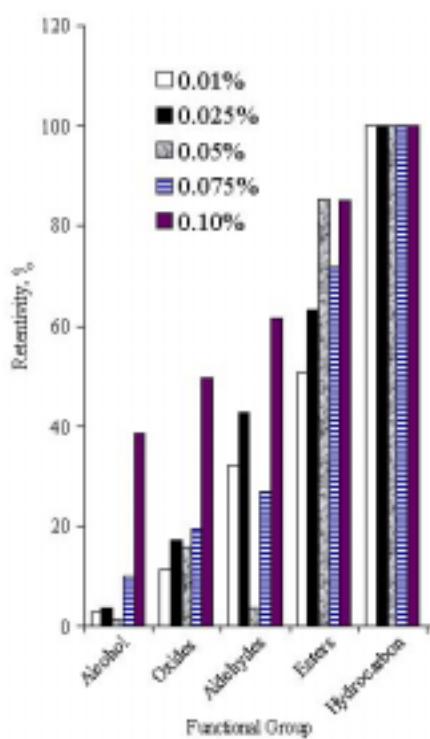
To investigate the effect of pectin concentration on the retention of the flavor compounds, the ultrafiltration experiments were carried out at pressure's value of 7 and 5 bar using 500,000 MWCO membrane. The flavor functional groups were plotted against the retentivity percentage for these groups. For an operating pressure of 5 bars pectin concentration of 0.1% showed the lowest retention for most of the components and this is due to the pectin layer that is created on the membrane surface. High pressure of 7 bar will push the layer against the membrane surface resulting in small amount of flavor compounds crossing the membrane. On the other hand at low pressure value of 2 bar, the pressure is not enough to transfer the flavor compounds across the membrane with the existence of the pectin layer. This result indicates that at high pressure the flavor permeation favored by low pectin con-

centration while at moderate pressure flavor permeation is not significantly affected by pectin concentration. The ultrafiltration experiment was conducted by injecting each one of the flavor components separately and the final results were plotted in Figures 3 to 4.

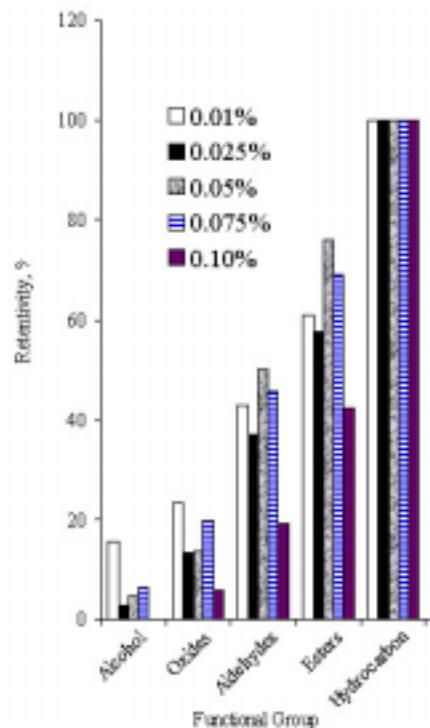
### Conclusion

The retention of the flavor compounds of guava juice is affected by the combined influence of applied pressure and pectin concentration, and this is due to the creation of the gel layer.

$\beta$ -caryophellene, which is considered an important compound in the flavor profile of the guava juice, had been adsorbed totally by the 100,000 and 500,000 MWCO polyethersulfone membranes because of its hydrophobic behavior. The molecule with the greater number of carbons in the alkyl group is more hydrophobic than a



**Figure 3.** Retentivity of the functional group at 7 bar using 500,000 MWCO membranes for different pectin concentrations.



**Figure 4.** Retentivity of the functional group at 5 bar using 500,000 MWCO membranes for different pectin concentrations.

molecule with fewer carbons in the alkyl group, and this would result in higher retentivity in the molecules with more carbons.

### **Acknowledgment**

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### **References**

Ferro R., Martha, L. and Castelblanco, R. 1969. Extraction and characterization of pectin from two varieties of guava (*Psidium guajava* L.) In "Chemical Abstracts", Vol. 71.48482b.

Heatherbell, D.A., Short, J.L., and Strubi, P. 1977. Apple juice clarification by ultrafiltration. *Confructa*. 22(5/6): 157.

Stevens, K. L., Brekke, J. E. and Sterns, D. J., 1970. Volatile constituents in guava. *J. Agric. Food Chem.* 18(4) : 598-599.

Sulaiman, N.M., Sulaiman, M.Z., and Yip, L.S., 1996. (August). Clarification of starfruit juice by membrane ultrafiltration, presented at the 12<sup>th</sup> International Congress of Chemical and Process Engineering, Praha, Czech Republic.

Wu, M.N., Zall R.R. and Tzeng W.C. 1990. Microfiltration and ultrafiltration comparison for apple juice clarification. *J. Food Sci.* 55:1162.

Yen, G.C., Lin, H.T., and Yang, P. 1992. Changes in volatile flavor components of guava puree during processing and frozen storage. *J. Food Sci.* 57:579.