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## Comparison of analytical methods for determination of limonoids in tangerine juice using HPLC-PDA and UPLC -ESI-Q-TOF

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Tangerine juices are still not much accepted by Brazilian consumers due its bitterness. The Murcott tangerine hybrids are more resistant to certain diseases and more appropriated to *in natura* exportation but the bitter taste is higher than in other varieties. In general, limonoids and flavonoids are the substances responsible for bitterness in citrus juices [1]. For flavonoids, the major agents of bitter taste are the flavanones naringin and glycosylated neohesperidin. The limonoids are limonin (main responsible) and nomilin [2], besides minor ones. The objective of this work was to evaluate the presence of limonin and nomilin (aglycones) in murcott tangerin pasteurized juice using HPLC-PDA and UPLC -ESI-Q-TOF. The method consists on extraction in an ultrasound bath for thirty minutes with acetonitrile as solvent. For HPLC method, the column was a Waters XTerra<sup>®</sup> (150 x 4.5mm; 5 $\mu$ m) and the mobile phase methanol: 0.1% formic acid 50%(v/v) at 1mL/min. The Chromatograph was a Waters Alliance<sup>®</sup> 2695 with PDA detector 2996 and extraction wave length at 210nm. The LC MS equipment was an UPLC-Q-TOF system. The inlet system used was an Acquity<sup>®</sup> UPLC and the ESI-Q-TOF mass detector was a Waters Synapt<sup>®</sup> MS G1. The chromatograph column was an Acquity UPLC BEH<sup>®</sup> C18 (5 x 2.1mm; 1.7 $\mu$ m), the mobile phase was methanol: 0.1% formic acid 45:55(v/v) at 0.2mL/min. The capillar energy was 4Kv. Collision cell energies were maintained at lowest level (4 kv) and the calibration error was 0.2198ppm. In the HPLC method, the standards of limonin and nomilin eluted at 6 minutes and 8 minutes, respectively, but no peaks were observed in samples extracts, despite several solvents and sample amounts were tested and bitter taste sensorial evaluated as very high. In a test with lemon juice a peak with low magnitude was observed. In the LC MS method, the extracted chromatogram showed for the standards and all studied samples a peak at 0.5 minutes with m/z 471.222 corresponding to protonated molecule of limonin and at 0.6 minutes with m/z 515.228 for nomilin protonated molecule. The MS analysis showed that conventional HPLC systems are not able to detect these substances in tangerine juice, probably due the low levels associated to a low selective work wave length (210nm). The HPLC work should continue with some sample concentration step to evaluate method recovery.

[1]MANNERS, G. D. *J. of Agricult. and Food Chem.*, **2007**. 55, 8285-8294.

[2] Vikram, A.; Jayaprakasha,G.K.; Patil, S. B. *Anal. Chim. Acta*, **2007**, 590 180–186.