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# Response Surface Methodology and UPLC-QTOF-MSE Analysis of Phenolic Compounds from Grapefruit (*Citrus × Paradisi*) by-Products as Novel Ingredients for New Antioxidant Packaging

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## ABSTRACT

The present work reports an experimental study on the optimization of phenolic compounds extraction from *Citrus × paradisi* peels and the development of active packaging. Response surface methodology (RSM) was applied to investigate the effect of microwave-assisted extraction (MAE) for the recovery of total phenolic compounds (TPC). The TPC in the optimized extract were 31.10 mg gallic acid equivalent/100 g of dry weight (mg GAE/100 g dw) with strong antioxidant capacity. The bioactive compounds in the optimized extract were determined by ultrahigh performance liquid chromatography coupled to quadrupole time-of-flight with high energy mass spectrometry (UPLC-QTOF-MSE). Forty-five compounds were detected and qualified. The optimized extract obtained by MAE was used as an active antioxidant in multilayer food packaging films. The multilayer low-density polyethylene (LDPE)/polyethylene terephthalate (PET) containing 10% of MAE optimized extract provided the most antioxidant power acting as a free radical scavenger.

**Keywords:** *Citrus × paradisi* peels, phenolics optimization, UPLC-QTOF-MS analysis, Antioxidant packaging material.

## 1 Introduction

This investigation aimed to examine the impact of diverse variables on the effectiveness and recovery of total phenolics (TP) extracted from *Citrus × paradisi* peel using the MAE procedure. In the present study, the response surface methodology was successfully employed to optimize total phenolic yield from dried *Citrus × paradisi* peels, according to different non-conventional solvent extraction processes (MAE). The extracts resulted from MAE and CE were incorporated into a multilayer LDPE/PET, which can scavenge the free radical from the package headspace. This active packaging represents the main novelty of this research

## 2 Experimental

The extraction parameters assessed were microwave power, extracting period and liquid-to-matter fraction to optimize the total phenolic amount. Beforehand, the bioactive molecules were identified and compared to those extracted by conventional extraction (CE). Once characterized, the extract was used as an antioxidant agent in new active packaging.

## 3 Results and discussion

### 3.1 Antioxidant activity of the extracts

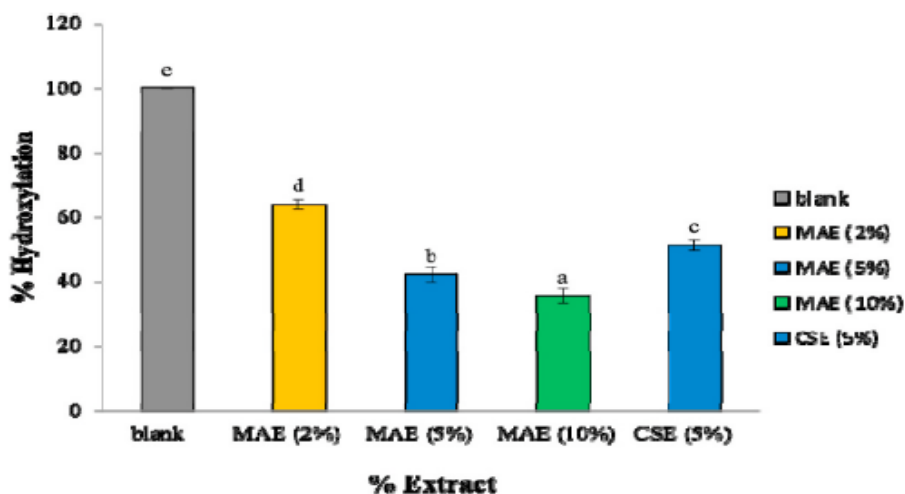
According to the results obtained, the extract obtained by MAE has a high antioxidant activity compared



to the one resulted from the conventional method (CE) in all the used tests. In DPPH scavenging capacity, the extract obtained from MAE showed significantly lower  $IC_{50}$  ( $12.81 \pm 0.14$  mg/mL) than CE method ( $20.89 \pm 0.41$  mg/mL). The same trend was noticed in ABTS assay, the extract from MAE has also significantly lower  $IC_{50}$  ( $1.90 \pm 0.02$  mg/mL) compared to CE ( $2.02 \pm 0.01$  mg/mL), but showed low scanning ability on ABTS radicals relative to other plant materials reported in the literature [16]. Besides, in the FRAP test, the significantly ( $P < 0.05$ ) superior activity was attributed to the extract from MAE with the  $IC_{50}$  value of  $3.43 \pm 0.15$  mg/mL compared to the one from CE with  $IC_{50}$  of  $7.16 \pm 0.17$  mg/mL. Citrus peel contain considerable amounts of highly active antioxidants [1]. According to the literature, the citrus skin antioxidant power is due to its richness in flavonoids, phenolic acids, and vitamin C [2]. The identified phenolics in these byproducts are hesperidin, narirutin, nobiletin, and tangeritin which are implicated in reduction of free radicals [3].

### 3.2 Antioxidant power of the elaborated packaging

The antioxidant capacity of films containing the two extracts applied into the bioactive packaging at different concentrations was measured, following the procedure developed by Pezo et al. 2006 [4]. The results obtained for the packaging subjected for twenty four hours to the ambience supplemented radicals produced using peroxide solvent. The results are stated in % of hydroxylation. This last diminish while the quantity of the MAE extract augments. There is a significant difference ( $P < 0.05$ ) between the different concentrations tested. The maximum antioxidant capacity was noted with film prepared with 10% of the MAE extract ( $35.28 \pm 2.36$  % of hydroxylation). Moreover, it demonstrated a significant diminution ( $P < 0.05$ ) comparatively to the blank (100%). The multilayer low-density polyethylene (LDPE)/polyethylene terephthalate (PET) containing 10% of MAE optimized extract provided the most antioxidant power acting as a free radical scavenger (fig.1).



**Figure1:** Antioxidant capacity of active film samples subjected to hydroxylation after 24 h.

The blank (plastic film without natural extract) is regarded as the 100% reference, MAE (Microwave-Assisted Extraction) (2,5 and 10%): the film contained the *Citrus × paradisi* peel extract at different percentages obtained by MAE method, CE (conventional extraction) (5%): the film contained the *Citrus × paradisi* peel extract at percentages of 5 obtained by CE method  $n = 3$ , a–e: Different letters indicates significantly different samples ( $P < 0.05$ ).

## 4 Conclusions

In this investigation, the adopted MAE procedure allowed a higher recovery yield of total phenolic compounds from *Citrus × paradisi* peels oxidant compounds in a limited period and used only small solvents. The extracts resulting from MAE and CE were incorporated into a multilayer LDPE/PET, where they were successful in scavenging free radicals, thus acting as strong antioxidants. Comparing the same

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concentrations (5%) of both extracts, the MAE extract gave the best results. Thus, the *C. × paradisi* by-products can be valorized by incorporating them into the food packaging films to improve their antioxidant power.

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