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Proceeding Paper

Recovering Antioxidant Compounds from the Juçara Residue Using a Green Approach †

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Abstract: Juçara (*Euterpe edulis*) is a Brazilian Atlantic Forest palm that produces phenolic-rich fruits used for pulp preparation. The pulp refining produces antioxidant and lipid-rich residue. This study applied glycerol as a green solvent for the microwave-assisted extraction of antioxidant compounds from residue. The variables included glycerol concentration (0–100%), microwave time (2–18 min), and temperature (53–187 °C). The total phenolic compounds (TPC) and DPPH• antioxidant capacity were influenced by glycerol concentration and temperature ($p < 0.05$). TPC ranged from 1052 to 5329 mg GAE/100g and antioxidant capacity from 29 to 191 µmol Trolox/g. The highest values were at 187 °C, using 50% glycerol for 10 min. All models were significant, with R^2 above 0.95. Glycerol, as a green solvent in microwave-assisted extraction, improves antioxidant compound recovery from juçara residue, presenting itself as a sustainable approach for adding value to the fruit production chain.

Keywords: *Euterpe edulis*; glycerol; microwave; extraction; Atlantic Forest

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1. Introduction

Juçara (*Euterpe edulis*) is a native palm tree to the Atlantic Forest, one of the main biomes of Brazil. To obtain its main product, the palm heart, the trees had to be felled, which caused juçara to be placed on the list of Brazilian species threatened with extinction. Alternatively, harvesting the fruits of the juçara palm tree does not involve cutting it down; thus, its utilization emerges as an alternative for species preservation. Furthermore, the use of the fruit should be seen as an opportunity for sustainable development using new technologies and research to promote the preservation of biodiversity and contribute to the development of activities that fortify local communities [1].

The main product obtained from the fruits is frozen pulp, a cheap and easy alternative to extend their shelf life. After the refinement of the pulp, a residue rich in bioactive compounds and macronutrients is obtained. Studies have already been conducted with this residue, aiming to increase the value of the juçara productive chain. Ribeiro et al. [2] obtained an antioxidant extract from juçara residue using ethanol as a solvent. However, variations in extraction operational conditions and the use of other solvents were not evaluated by these authors. To reduce the environmental impact of the residues and the extraction process, green solvents and cleaner extraction techniques are becoming more important, such as the use of glycerol associated with microwave heating [3].

Glycerol is a byproduct of biodiesel production. With the increasing use of biodiesel as a replacement for fossil fuel-based fuels, glycerol becomes an abundant byproduct in the

market. Concerning its characteristics, glycerol exhibits a series of interesting attributes that justify its application as a green solvent, such as being non-flammable, miscible in water, having low volatility, and low cost. However, its use as a solvent in the extraction of bioactive compounds from biomass has been relatively unexplored, partly due to its viscosity. To increase the efficiency of extraction, binary solvent systems can be employed by combining glycerol and water, as well as using high temperatures. However, the main advantage of glycerol that supports its application as a solvent is itsnon-toxicity since it is classified by the FDA as GRAS, meaning that it is a substance recognized as safe. This feature allows for the direct use of extracts in formulations without requiring purification or fractionation steps [3–6].

The use of microwaves as an extraction technique offers advantages such as shorter processing time, automation, and reduced solvent consumption. Furthermore, its application in extraction is efficient due to increasing pressure inside the plant's matrix cells, leading to the rupture of the cell wall and releasing easier target compounds [7,8].

Therefore, this study aims to evaluate the use of glycerol as a green solvent in the microwave-assisted solid–liquid extraction of the antioxidant compounds from juçara residue.

2. Materials and Methods

2.1. Samples

The juçara residue used in this study was obtained from juçara pulp centrifugation performed on the pilot plant of Embrapa Agroindústria de Alimentos (Guaratiba, Rio de Janeiro, Brazil). The residue was defatted and dried before antioxidant recovery. This material exhibited 59.46% of particles with a size range between 0.297 mm and 0.590 mm.

2.2. Microwave-Assisted Solid–Liquid Extraction

Glycerol, water, and their combinations were used as a solvent, along with varying temperatures from 53 to 187 °C and processing times from 2 to 18 min, as shown in Table 1. The combination of independent variables (solvent, temperature, and time) was established via experimental design, using a rotational central composite comprising 17 experiments. The extracts were subjected to microwave treatment (Ethos 1, Milestone, Sorisole, Italy), and the solid–liquid proportion was fixed at 1:30 (w/v) for all experiments. After each process, the extracts were vacuum-filtered and stored frozen or subsequent experiments.

Table 1. Experimental design for antioxidant compounds recovery from juçara residue and results obtained for TPC and DPPH•.

Experiment	Glycerol (%)	Temperature (°C)	Time (minute)	TPC (mg GAE/100 g)	DPPH• (µmol Trolox/g)
1	20	80	5	1083	31
2	20	80	15	1052	30
3	20	160	5	3208	90
4	20	160	15	3228	98
5	80	80	5	1151	30
6	80	80	15	1587	40
7	80	160	5	4311	140
8	80	160	15	4222	134
9	0	120	10	1448	41
10	100	120	10	2478	63
11	50	53	10	1096	29
12	50	187	10	5329	191

Table 1. *Cont.*

Experiment	Glycerol (%)	Temperature (°C)	Time (minute)	TPC (mg GAE/100 g)	DPPH• (µmol Trolox/g)
13	50	120	2	2026	68
14	50	120	18	2447	90
15 (CP)	50	120	10	2711	72
16 (CP)	50	120	10	2388	64
17 (CP)	50	120	10	2286	61

CP—Central Point; GAE—Gallic Acid Equivalent.

2.3. Determination of Total Phenolic Compounds (TPC)

The TPC content was evaluated using the Folin–Ciocalteu reagent, following the method described by Singleton and Rossi [9]. In the method, 250 µL of the extract was mixed with 1.25 mL of 10% (w/v) Folin–Ciocalteu reagent (Imbralab, Ribeirão Preto, Brazil). After 2 min, 2 mL of 7.5% (w/v) sodium carbonate solution was added. The reaction proceeded for 15 min at 50 °C, and the tubes were subsequently cooled in an ice bath. The absorbance was measured with a spectrophotometer (SP 220, Biospectro, Curitiba, Brazil) at 760 nm using ultrapure water as blank. For quantification, a calibration curve was prepared using gallic acid (Sigma-Aldrich, Saint Louis, MO, USA) as the standard, which ranged from 10 to 100 mg/L. The results were expressed as mg of gallic acid equivalent per 100 g sample (mg GAE/100g).

2.4. DPPH• Assay

The antioxidant capacity using the DPPH• radical method was determined as described by Hidalgo [10]. In this method, 100 µL of the extract was mixed with 2.9 mL of DPPH• solution at 6.0×10^{-5} M (Sigma-Aldrich, Steinheim, Germany), which was diluted with methanol to obtain a solution with absorbance between 0.600 and 0.800. The reaction took place for 30 min after the addition of the DPPH• radical. The absorbance of the solution was measured with a spectrophotometer at 517 nm, with methanol as the blank. A calibration curve was constructed using Trolox standards (Sigma-Aldrich, Buchs, Switzerland), and the results were expressed as µM of Trolox per gram (µM Trolox/g).

2.5. Statistical Analysis

The experimental data obtained were evaluated on Statistica software version 13 (Dell Inc., Tulsa, OK, USA) viaanalysis of variance (ANOVA) and Pareto chart. The test to determine the model's lack of fit and determination of R^2 were employed to verify the model's significance, considering a confidence interval of 95%.

3. Results and Discussion

Table 1 presents the results obtained from the conditions proposed by the experimental design. It is possible to observe that the independent variables influenced the TPC content of the extracts, ranging from 1052 to 5329 mg GAE/100 g (experiments 2 and 12, respectively). The same can be applied to the antioxidant capacity determined by the DPPH• method, with the best result being 191 µmol Trolox/g, which was also observed in experiment 12. These data indicated a significant influence caused by the increase in glycerol concentration on the extraction solution, as well as temperature, once experiment 12 employed 50% glycerol as the solvent and a temperature of 187 °C, which were both higher than the levels used in condition 2. It is important to emphasize that the TPC content and antioxidant capacity by DPPH• observed in this study are superior to those found by Ribeiro et al. [3], which reported a content of 1116 mg GAE/100 g and 39 µmol Trolox/g, respectively, for juçara residue extract obtained with 30% of ethanol at 70 °C for 1 h. This

corroborates the efficiency of the extraction system using glycerol as a solvent in microwave-assisted extraction.

The Pareto chart presented in Figures 1 and 2 indicates that the TPC and antioxidant capacity of the extracts were influenced mostly by temperature and glycerol concentration in the extraction solution, with positive linear effects for both independent variables. For the temperature variable, the quadratic effect was also significant, meaning that high temperatures can reduce the antioxidant potential of the extracts, possibly due to degradation or changes in the structure of the extracted compounds. Also, it is possible to observe that the variable time did not influence the evaluated responses.

The influences of the independent variables observed in this study can be explained via the thermodynamic aspects involved in extraction processes. According to Oliveira [11], increasing the temperature facilitates the extraction of phenolic compounds by increasing the solubility coefficients and the diffusion of compounds to be extracted while also reducing the viscosity of the solvents. Furthermore, it is known that the polarity of the solvent is crucial for the efficiency of bioactive compound extraction processes. For example, low-polar solvents extract smaller amounts of phenolic compounds, resulting in extracts with lower antioxidant potential [11]. Thus, the glycerol/water mixture provides adequate polarity to the system and reduces the viscosity of pure glycerol, explaining the high values of bioactive compounds and antioxidant capacity in the present study compared to the data reported by Ribeiro et al. [2].

In terms of the statistical analysis, the models were significant in predicting the behavior of the responses concerning the independent variables, as the calculated F-values were higher than the listed F values ($F_{9,7} = 3.68$) at $p = 0.05$. For TPC content and antioxidant capacity using the DPPH• method, the calculated F values were 117 and 38, respectively. Moreover, it is worth mentioning that the lack of fit of the models was not significant, as it presented p-values higher than 0.05, and the calculated F values were lower than the listed F values for all responses. The R^2 values of the adjusted models were 0.98 and 0.95 for the TPC and DPPH•, respectively, indicating that the models explained at least 95% of the variability in the data obtained from the experimental design.

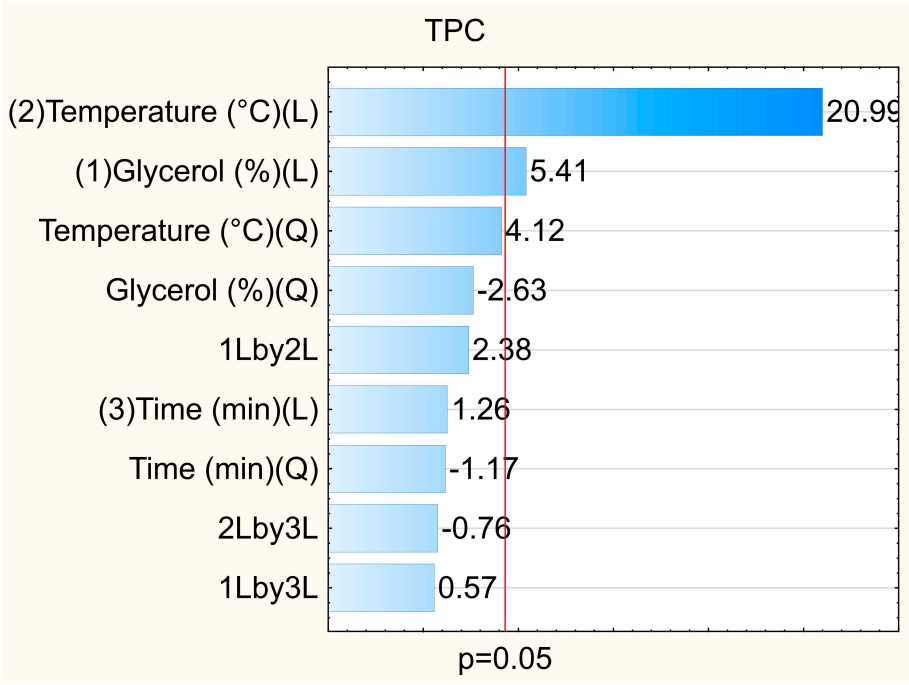

Figure 1. Influence of independent variables on the TPC content of the extracts.

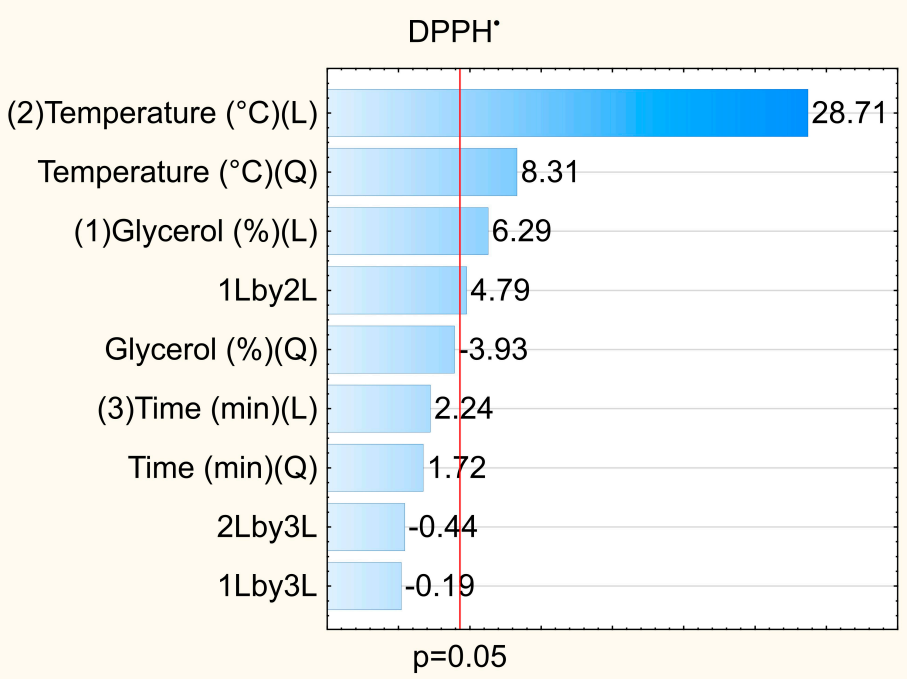

Figure 2. Influence of independent variables on the antioxidant capacity via the DPPH• method.

4. Conclusions

Considering the results obtained, it can be concluded that the residue from juçara pulp refinement can be used as a raw material to obtain bioactive compounds. Additionally, the use of glycerol as a solvent in microwave-assisted extraction favored the recovery of these compounds, emerging as a promising approach to add value to the agro-industrial chain of the juçara fruit in a sustainable way.

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