Development and optimization of a microwave-assisted extraction process of spent coffee grounds using betaine-glycerol Natural Deep Eutectic Solvent

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Introduction

Coffee, with a global consumption of 2.25 billion cups per day, is one of the mostly consumed nonalcoholic beverages in the world. It is estimated that 650 kg of spent coffee grounds (SCG) are generated during the processing of 1 ton of green coffee which mainly disposed in landfills. However, SPG, consist a valuable source of bioactive compounds and so, their valorization is of great interest. The choice of the appropriate extraction method of bioactive compounds is one of the most important factors in the raw material process utilization. Natural deep eutectic solvents (NADES) are currently emerging as a new class of green solvents that are effectively used as alternative extraction media of bioactive compounds from raw natural materials and biomass.

In this context, in this work, it is presented a green approach for the efficient extraction of bioactive compounds from SPG. Microwave Assisted Extraction (MAE) method was used as a high energy technique in combination with the betaine/glycerol NADES, in order to simultaneously minimize the extraction time and maximize the extraction yield. For comparison reasons, extractions using hydroethanolic solutions as extraction solvents were also implemented. The proposed methodologies were optimized by performing Experimental Design (Taguchi method) to study the effect of time, SPG-to-solvent and NADES/water ratio on the total phenolic content (TPC) and flavonoid content (TFC).

Experimental

Sample preparation: All experiments were performed using espresso coffee residual of no specific variety collected from a domestic machine. Optimized conditions were applied on different varieties (Arabica, Kenya, Guatemala, Ethiopia, Brazil and Ruanda). The SPG were air dried for 72 hours in a dark room and kept at 4°C.

Conventional extraction was performed using ethanol/water mixtures in varying ratios. The Solid-to-Liquid ratio was 3% in all cases and the samples were stirred on a magnetic stirrer for 24 hours at ambient temperature. MAE was performed using the Start D apparatus of Milestone. An appropriate quantity of SPG was mixed with a certain volume of solvent in order to achieve the required Solid-to-Liquid ratios according to the experimental design.

Taguchi Design Of Experiments (DOE): Four parameters with values of three different levels were studied (L9 orthogonal array) in order to study their effect on TPC and TFC of the extracts.

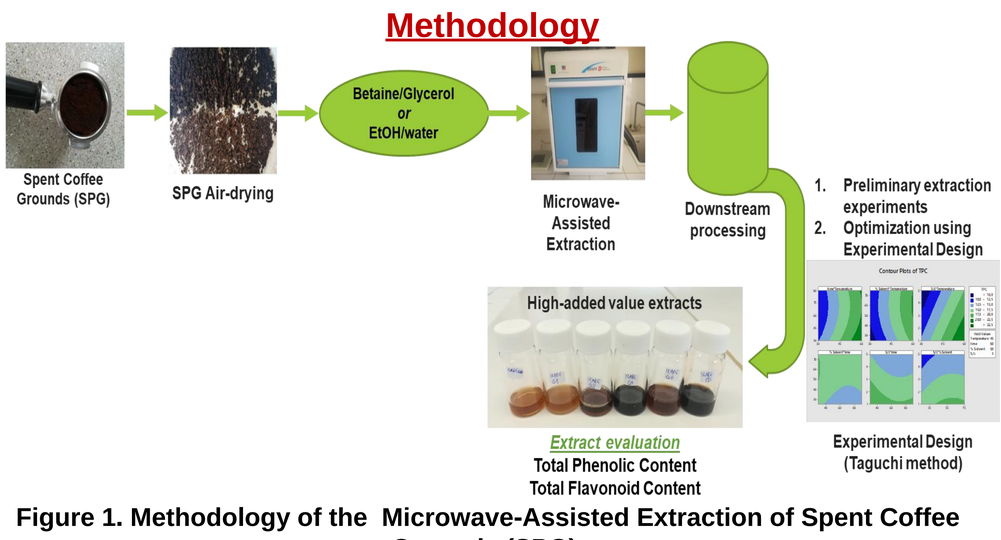

Figure 1. Methodology of the Microwave-Assisted Extraction of Spent Coffee Grounds (SPG).

Conventional extraction of SPG

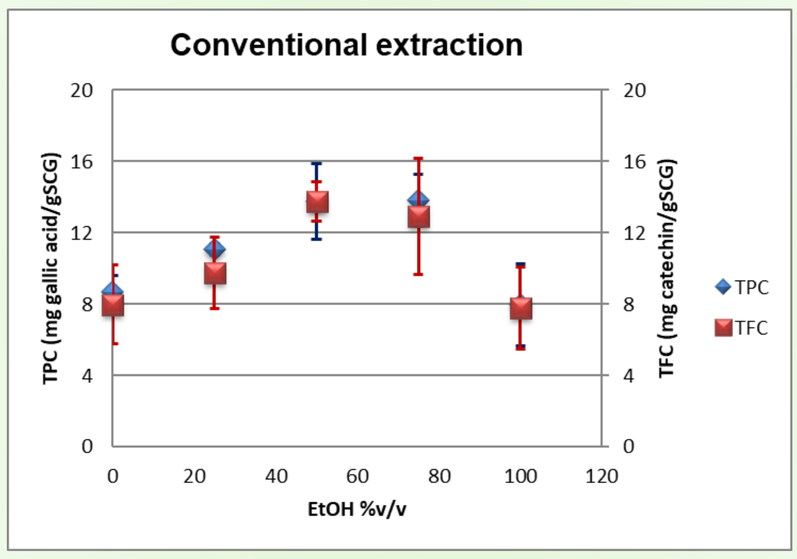

Figure 2. Conventional extraction of SPG using EtOH/Water systems of various concentrations (Conditions: 24h stirring at ambient temperature, Solid-to-Liquids ratio: 3% w/v)

Design Of Experiments (DOE) for the MAE of SPG

Table 1. Conditions of the experiments indicated by the Taguchi DOE.

Temperature (°C)	Time (min)	NADES or EtOH -to-water ratio (%v/v)	Solid-to-Liquid ratio (% w/v)
30	30	25	1
30	60	50	3
30	90	75	5
45	30	50	5
45	60	75	1
45	90	25	3
60	30	75	3
60	60	25	5
60	90	50	1

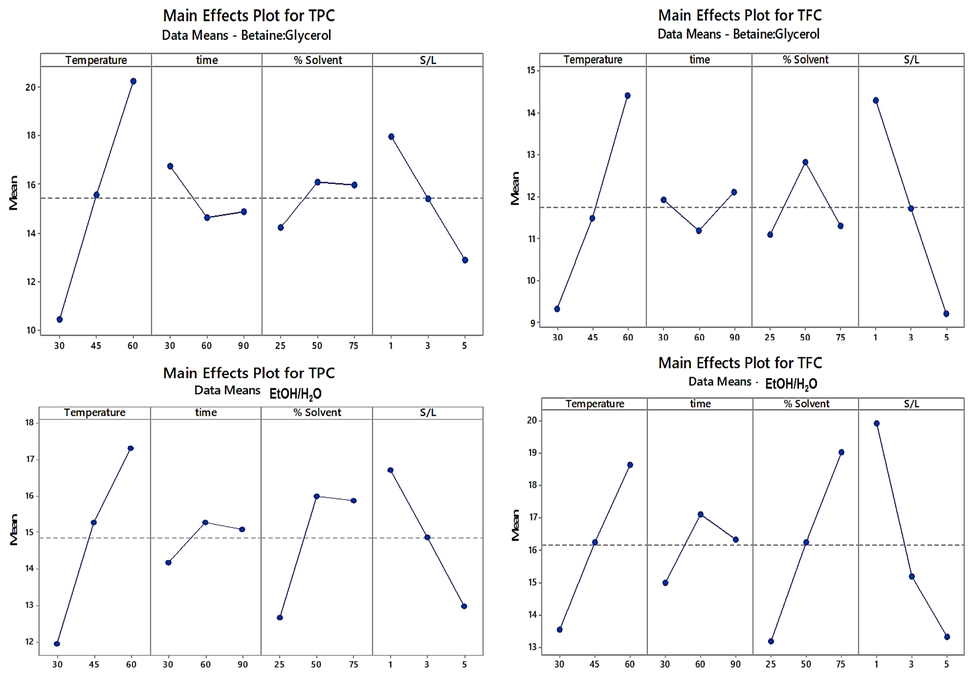

Figure 3. Main Effects Plot for TPC and TPC for the extractions using the NADES betaine-glycerol (up) and the hydroethanolic solution (down)

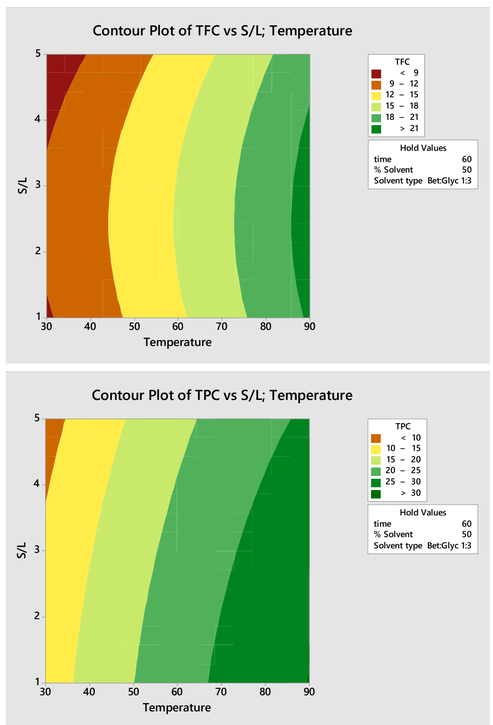

Figure 4. Indicative Contour Plots of TFC (up) and TPC (down) vs Solid-to-Liquid and Temperature parameters.

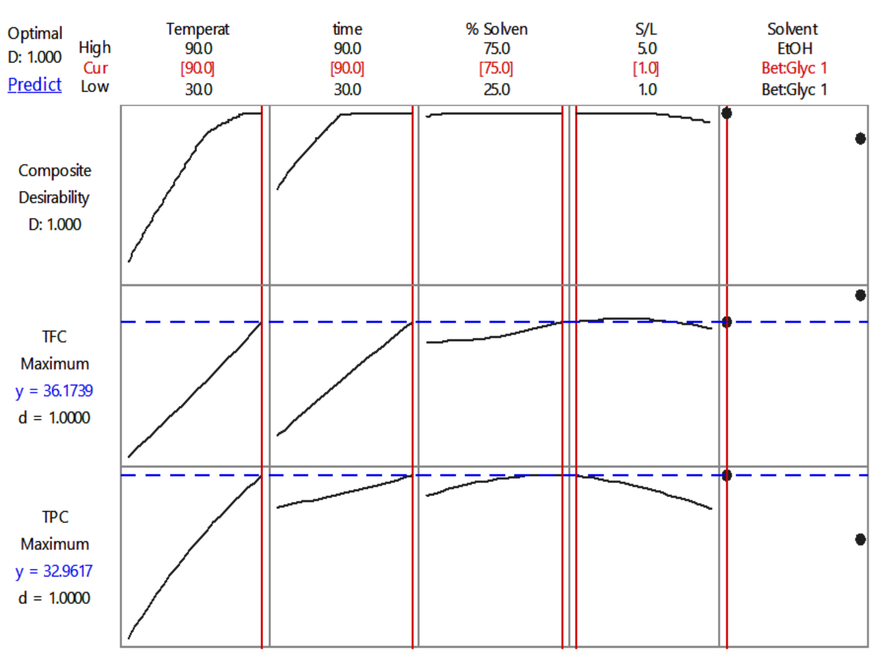

Figure 5. Optimization results from the models derived from the Taguchi DOE

Table 2. TPC and TFC of the extracts of different coffee varieties from MAE with NADES/Water 50:50v/v, 1% solid-to-liquid ratio at 90°C for 60 min.

Coffee variety	TPC (mgGAE/gSPG)	TFC (mgCATE/gSPG)
Kenya	29.8±2.5	20.4±3.3
Ethiopia	21.5±0.2	15.8±0.6
Guatemala	21.2±2.3	18.8±1.5
Brazil	19.9±2.9	16.2±1.8
Ruanda	22.1±1.8	17.1±2.1

Conclusions

- ✓ Valuable phenolic and flavonoid compounds were extracted from **SPG** were extracted using a the **NADES** consisting of **Betaine and Glycerol** as well as using **hydroethanolic solutions.**
- ✓ The one way ANOVA performed on all results obtained by both solvents, reveals that the most critical parameters for TPC and TFC are **temperature** and **Solid-to-Liquid ratio**.
- ✓ The **optimum solvent** was found to be the **NADES** according to the Taguchi experimental Design implemented for both solvents .
- ✓ The **optimum conditions** are**: 90 ˚C, 90 min, 1% w/v Solid-to-Liquid ratio, 75% v/v NADES-to-water ratio.**