Design of a prototype antioxidant isolation unit from espresso coffee beverage production waste

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Abstract

The objective of this study was the optimization of the recovery process for the isolation of phenolic compounds from coffee spent grounds in series of extraction experiments in the laboratory. The optimal parameter values from extraction experiments were used for the design of the prototype pilot unit. The following parameters were investigated: solid to solvent ratio (g waste/5-50 mL solvent); ethanol percentage in the mixture of the solvent (0-100%); extraction time (5-360 min); mixing rate (50-250 rpm) and use of ultrasound to enhance the release of organic compounds from the matrix of solid wastes. Additional experiments dealt with the effect of consecutive extractions on the final recovered amount of phenolic substances, as well as with the imposition of the final optimal sample in rotary evaporation. The parametric study showed that the optimum process for high phenolic extraction were: 1 g of solid per 10 mL of solvent (wet solid/solvent ratio =1:10), 20 mL H_2O per 80 mL EtOH (EtOH solvents' ratio, 80%), extraction time equal to 60 minutes, ultrasound applied for 10 min and an optimum number of consecutive extractions equal to one. The final percentage for the purity of phenolic compounds comparing only with carbohydrates concentration was up to 60%. Based on the optimal condition values, a technoeconomic study and a pilot unit design took place. Initially, the process flow diagram was constructed, and the technical characteristics and dimensions were analyzed. Then, the cost of the equipment was estimated to examine the financial viability of the pilot unit. The unit capacity was set at 12 t/cycle for 40 cycle/year and the indicative selling price is not yet identify because of the variety of phenolic product on the market. Based on the total annual income and production costs, the tax disbursements, the loan interest, and depreciation we concluded that, through a 6-year long investment plan, the proposal is profitable.

Keywords: Spent coffee grounds, Phenolic compounds, Solvent extraction, Pilot unit, Techno-economic study

Introduction

The coffee is the most popular beverage in the world and has occupied the research community in recent years mainly because of its beneficial properties, but also because of the huge quantities of wastes created by the coffee manufacture [1,2]. Each year it is estimated, that about 6 million tons of coffee wastes are discarded, particularly in landfills [3,4], suggesting that there is an urgent need for immediate management. The uncontrollable discharge of wastes into the environment creates hazardous problems because of their high organic load and the presence of toxic compounds for plants, such as caffeine and tannins, while their combustion burdens the environment. Alternatively, recovery of phenolic substances and of other organics [5,6] from the solid wastes could neutralize the toxic effect of the final discarded residuals. Phenolic substances are compounds of high added value that exhibit antioxidant and antimicrobial activity. Phenolics, protect the human organism from chronic degenerative diseases such as cancer and disease Alzheimer 's and can be used in food, drug and cosmetic industries [7,8]. The economic value of phenolic compounds in the market depends on their purity and the presence of hydroxy acids in benzyl hydrocarbons, which are responsible for their antioxidant properties. Laboratory experiments were performed for extraction of phenolics from residuals from ' bulk ' coffee (spend coffee ground, SCG) as well as from spent coffee wasted in capsules (SCGc). The parametric study illustrated that processing of SCG showed better results in terms of recovering of phenolic compounds than in the process of SCGc and this seems to be in line with the literature [9,10]. However, it should be noted that although phenolic compounds are in higher concentrations in SCG, the literature states that the antioxidant effect of phenolic compounds obtained from the SCGc may be more important [9]. Another important factor that should be considered is the content of coffee residuals, which varies depending on the variety of coffee beans, i.e. if it is a variety of Arabica, Robusta or a mixture of them [10-13]. Using the optimal parameter values for extraction of phenolic substances, a preliminary design of a unit was made for capacity equal to 480 tn/y. Next, a financial study considering the fixed and operating costs of the plant was performed.

Experimental

For the experimental study, samples were used with regular coffee residues (SCG) and samples of coffee residues from capsules (SCGc) as well, with different solid proportions in liquid solvent (1:5, 1:10 1:20 and 1:50). The used solvents were distilled water, pure alcohol (95%) and mixtures of alcohol/water in various proportions.

The extraction was performed under continuous stirring at ambient temperature and selected temperature values (range 25-80^{o}C), on a tabletop stirring jar tests device, Raypa®. In most experiments, the duration of extraction was 60 minutes which ensured the maximum possible recovery. This extraction duration was chosen after tests at different time intervals: 15, 20, 45, 60, 90 and 120 minutes. Optimum stirring rate was found equal to 150 rpm after testing at a range among 50-200 rpm. At the end of the extraction, the solid residuals were removed, and extract solutions were stored in the refrigerator at 4^{o}C, in glass bottles for subsequently measurements of the total phenolic compounds and total carbohydrates.

The measurement of phenolics was performed using the known method Folin-Ciocalteu [14]. The calibration was done with standard gallic solutions at 760 nm in a UV spectrophotometer. Next, carbohydrates were measured using L-Tryptophan reagent and measurements were done in a portable photometer (Smart 3 colorimeter, LaMotte®). Absorption values were measured at 525 nm, while concentrations of carbohydrates were estimated with a standard solvent based on tryptophan. The ratio of phenolics over carbohydrates is very important for the development of financial analysis because this ratio defines the purity of the isolated samples. The purity of the isolated solutions is an index for the antioxidant strength of the purified compounds. According to this ratio and after the financial analysis the price of the product can be set, and check if the suggested pilot unit is profitable, so to continue with a more detailed analysis.

Result and Discussion

The extensive parametric study included several series of experiments, however in the present work, only characteristic experiments from the treatment of the two types of residuals at different concentrations of solid/liquid solvent are included.

Comparison of phenolics concentrations in SCG and SCGc

Two samples of normal brown residues (SCG) and two samples of coffee residues from capsules (SCGc), were tested in the beginning. The solid-liquid ratio was selected to be 1:10, as it was found as the best proportion, in terms of extraction of phenolic substances, according to previous laboratory experiments. The best solvent was not pure ethanol but a solvent of 400 ml water/ethanol in a ratio of 1:4 (320 ml Ethanol and 80 ml Water supply).

The obtained results for the recoverable quantities of phenolic substances and carbohydrates for the two types of residues are shown in Figure 1. The experiments were repeated three times and slight differences among them were detected.

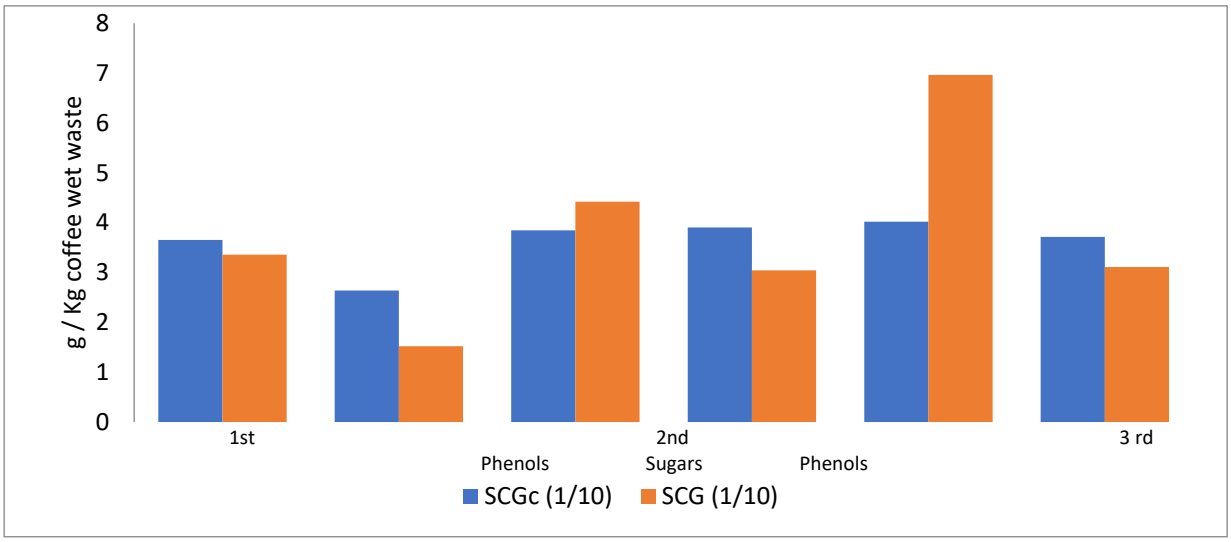


Figure 1: Recovered quantities of phenolic substances and carbohydrates in 3 different extraction experiments for the two types of coffee residues.

The purpose of this study was to obtain phenolic compounds at the maximum possible concentration and the lowest possible concentration of carbohydrates. The purity of the final product calculated based the concentration of phenolics and carbohydrates only. On future experiments all substances in the final product will be analyzed with HPLC process. In the 1st experiment, it is observed that the amount of phenolics in SCGc is 8.7% higher than that of the phenolics in SCG. However, carbohydrate concentration in 1st experiment is greater by a factor of 73% than that in SCG. In the 2nd experiment, phenolic concentration in SCG is 15% higher than in SCGc while carbohydrates in SCG and less by a factor of 28.3% than in SCGc. In the 3rd experiment, there is a considerably greater amount of phenolics in SCG (73.2%) than in SCGc while carbohydrates in SCGc are 19.4% higher than in SCG. In summary, the total series of experiments revealed that SCG gave better results, up to 5 gr of phenolic substances. This shows two possible scenarios: the first possible scenario refers to the case where the coffee (or the variety of coffee) that used in coffee shops as a 'bulk' product contains more phenolic substances. The second scenario is that the extraction of phenolics from SCGc with hot water/steam at high pressures is more effective and more organic substances are extracted in the beverage, including phenolic compounds. Thus, less amounts of phenolic substances were left in the residual. A pleasant remark is the low carbohydrates concentrations in all 3 experiments where the average value is around 3 gr per kg of solid coffee residue. Significant differences in the values of carbohydrates were not observed for the two types of wastes.

Finally, according to SCGc (capsule of Nestle®) experiments, constant amounts of phenols and carbohydrates were observed in all samples, and this supports the hypothesis of a pretreatment process of the coffee before it is used to produce espresso capsule.

The optimal ratio of liquid solvent to the solid mass.

A parameter which has been thoroughly examined in this work was the ratio of the mass of the solid residual to the volume of the solvent, in order to recover the maximum possible phenolic substances contained in the solid matrix of the residual. The ratio for solid/liquid were 1:5, 1:10, 1:20 and 1:50, based on the hypothesis that in large quantities of solvent, phenolic compounds that are trapped in the solid structure of residues will be recovered more readily. Figure 2 shows the quantities of phenolic substances recovered for the two solid/liquid ratio, 1:10 and 1:50 ratio values since ratios of 1:5 and 1:20 didn't show any significant differences with the organics obtained in the ratio of 1:10.

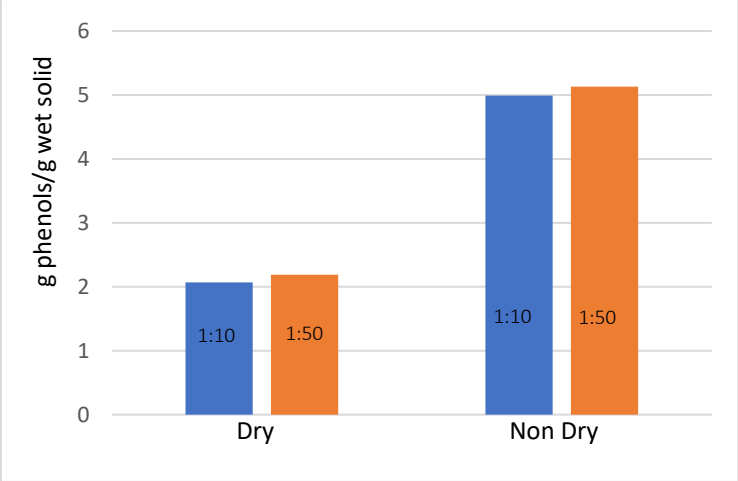


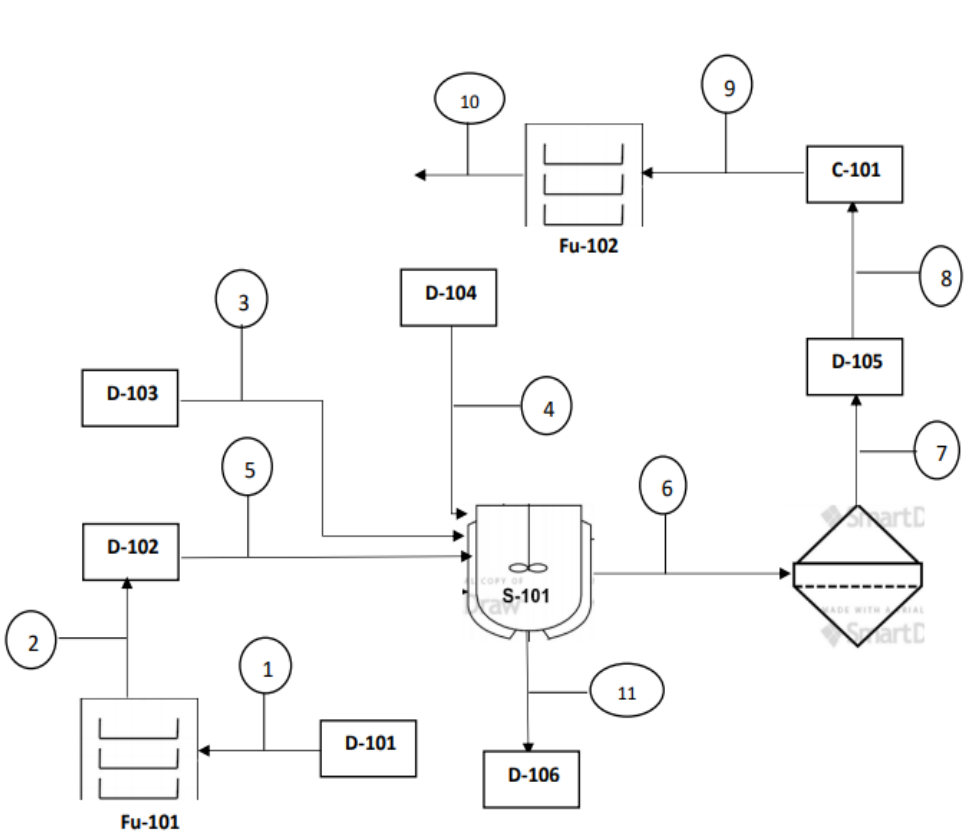
Figure 2: Quantities of phenolic substances extracted in various solid/liquid ratios with solids a) Dried at 105°C b) with their initial moisture content (68%)

Figure 2 b shows that both ratios (1:10 and 1:50) gave the same results in terms of recovered phenolics which means that a volume of 10 mL per gram of residual is enough for the maximum recovery of phenolic compounds. Figure 2a shows results for the recovery of phenolic compounds in samples that were dried at 105°C overnight. The measurements taken from the extraction of the two types of solids were normalized by the weight of the raw waste in order direct comparison between them to be possible. Figure 2a suggests that the effect of moisture is important for the phenolic recovery. It is evident that the quantities of extracted phenolic compounds, are higher if the moisture has not been removed from the original waste for both solid-liquid ratios (1/10 and 1/50).

Financial analysis:

After the optimization of the conditions for the extraction of coffee residues (SCG), a financial analysis was carried out in order to find the changes in the investment and the production costs, for different processing quantities of coffee residuals per year and different facilities per unit operating cycle. The pilot unit (Figure 3) consists of storage tanks for the SCG, H_2O and EtOH, a tank for the extracts, a rotary evaporator, membranes, refrigeration drying equipment, microwave oven, and other auxiliary equipment.

The original value of the quantity SCG per year in Greece is 500 ton. If the unit operates at 300d/y and that the total benefit is 12 tn/operation cycle, the duration of each cycle is 6d. If for the calculations we use an average quantity value of the substance extracted around 50 g/Kg, then 24 tn/y of phenolic solutions can be produced with 60% purity comparing only phenols and carbohydrates concentrations. The calculations for fixed and operating costs for different capacity values are shown in Figures 4a and b.



D-101 Waste tank before dry

Fu-101 Dryer

D-103 Water tank

D-104 EtOH tank

D-102 Waste tank after dry

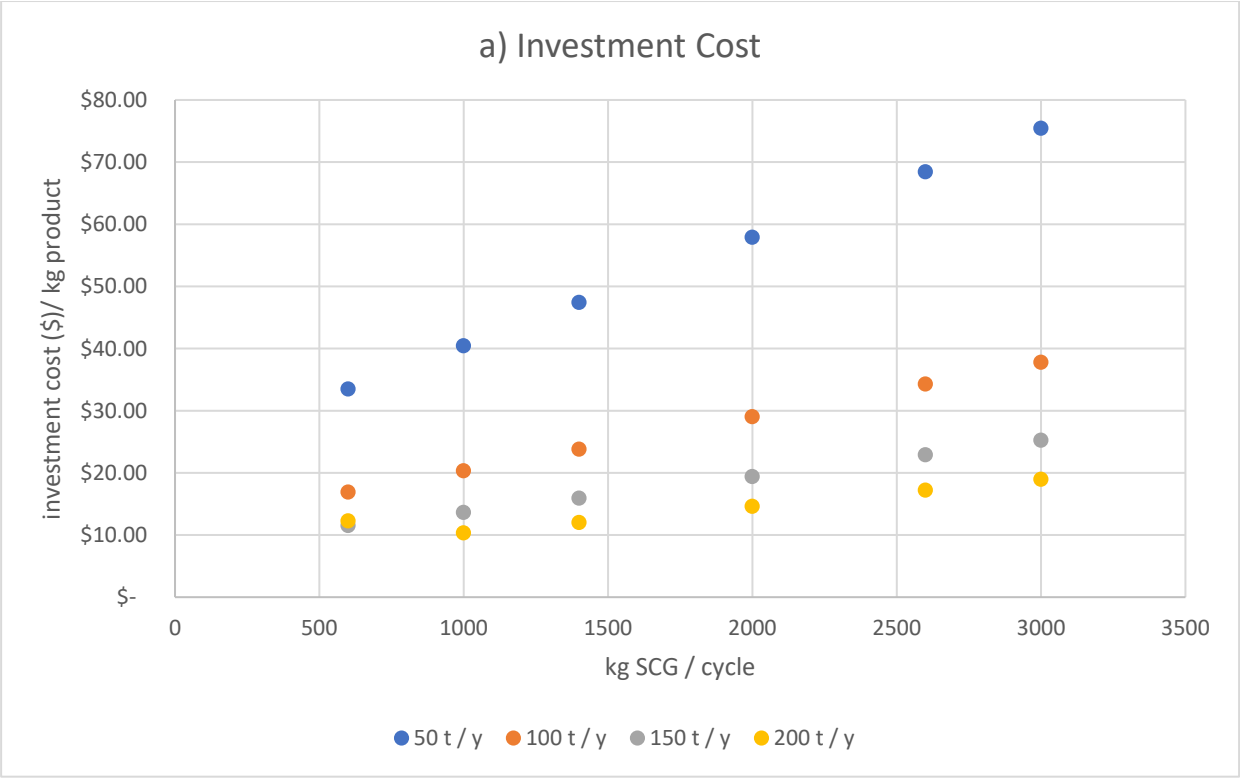
S-101 Extraction tank

D-105 Stabilizes tank

C-101 Evaporator

Fu-102 Freeze dry

Figure 3: Process flow diagram



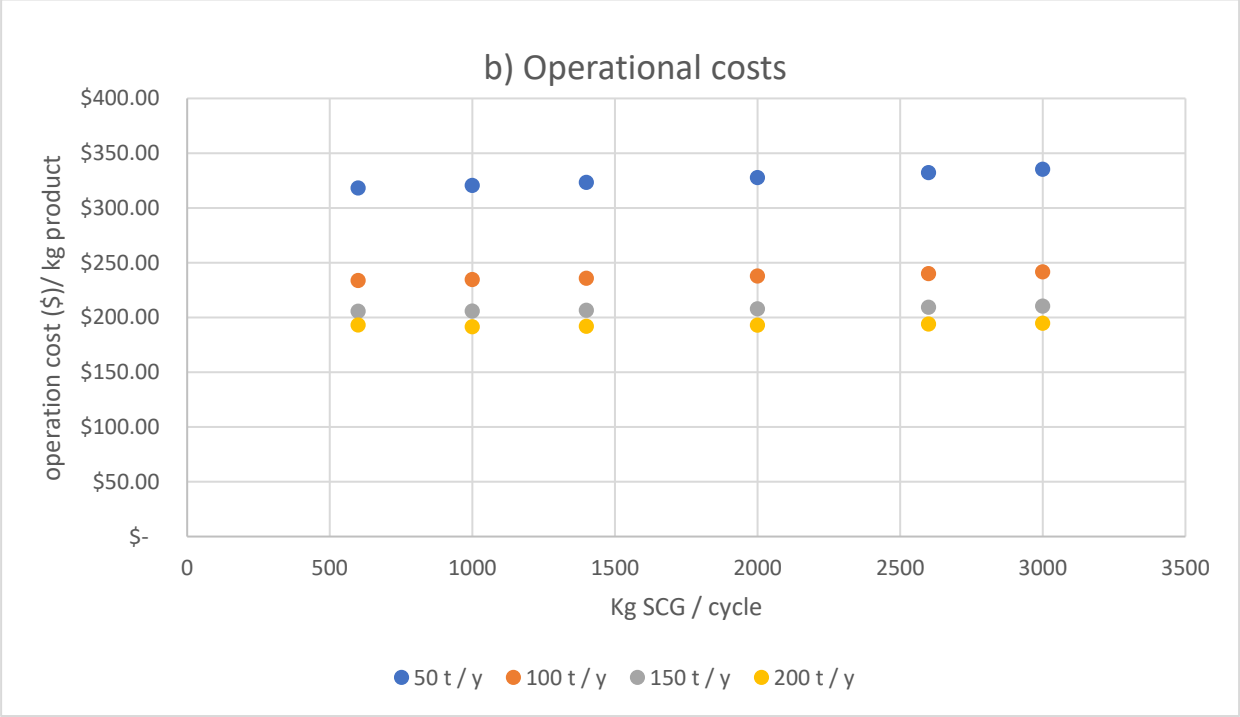


Figure 4: a) investment cost for various capacities of the unit, b) operating costs for various capacities of the Unit

As shown in Figure 4a, it is observed that generally, as the volume to be processed per cycle is increased then the cost of the initial investment is increased, as expected. But, as the processing volume per year increases, the slope of the investment cost is lower. Thus, at high capacities the investment cost can be reduced (per kg of product). On the contrary, in Figure

4b, it appears that the processing volume per cycle isolation does not affect the operating costs, but the increase in the processing volume per year reduced operating costs.

Conclusions

For the isolation of phenolic antioxidants from agro-industrial waste, it is necessary to examine the optimal conditions and find the best parameter values for the isolation in combination with the most economical sets of procedures. Economic analysis is needed to reveal the feasibility for the transition of the process from laboratory to industrial case. Almost all agro-industrial wastes can be considered as a source of natural antioxidants, but few of them can be considered as economic sources of phenolics. The present work on the extraction of phenolics from coffee waste espresso wastes showed that an industrial unit is possible to be designed and economic profit is expected. Optimal conditions were found for the recovery of phenolic compounds with very good purity up to 60% of antioxidant substances comparing with carbohydrates. According to the financial study, as the processing volumes increase the investment cost increases, but the rate is smaller at high volumes. On the other hand, the operational cost remains constant. Thus, it was observed that for the largest scale of 200 tons per year, processing costs can be less than 200 euros per kilo of waste, which is close to the market price. In the future, we will have full analyses the final product so to calculate the accurate purity of phenolic extraction. So, coffee waste espresso can be considered for industrial processing after an extensive market study and a detailed operational plan must be carried out.

Acknowledgments

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