INTEGRATED PROCESS DEVELOPMENT AND TECHNO-ECONOMIC OPTIMIZATION FOR THE VALORIZATION OF INDUSTRIAL POTATO PEEL WASTE

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ABSTRACT

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Integrated Process Development and Techno-Economic Optimization for the Valorization of Industrial Potato Peel Waste

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One third of produced food is discarded, along with millions of tons of potato peel waste. As the peels contain bioactive polyphenols used across several industries, their recovery has increasingly been studied to reduce market prices and improve potato processors' profitability. While current extraction studies either neglect techno-economic feasibility or lack process development and optimization, this Thesis compared novel ultrasound-assisted extraction (UAE) and conventional heat reflux extraction (HRE) along with two pretreatment options (wet, dry), and optimized the process for techno-economic performance. Design of experiments coupled with techno-economic analysis optimized the key factors affecting profitability and selectivity as follows: drying and milling pretreatments, two HRE steps with 10-min residence time each at 60 °C, L/S ratio of 8 ml/g, and 50 % ethanol-water solvent. After a washing step, the process recovered 96 % of extractable polyphenols, comprising 0.73, 0.16, and 0.76 mg/gdb of chlorogenic acid, caffeic acid, and α-chaconine, with a total phenolic content of 4.82 mgGAE/gdb. The estimated production cost was 12 €/$kg_{extract}$. Low L/S ratio was identified as critical for process economics, while a biorefinery approach, producing polyphenols, biochar, and fertilizer, may further improve viability. UAE remained less practical, requiring further optimization against techno-economic indicators and selectivity. Although pilot experiments and purification process development remained uninvestigated, the designed process appeared technically and economically feasible and could therefore recover valuable components from industrial potato peel waste while simultaneously contributing to the circular economy and reduction of food waste.

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Kolmasosa tuotetusta ruoasta hävitetään, mukaan lukien miljoonia tonneja perunankuorta. Koska kuoret sisältävät useilla eri teollisuudenaloilla hyödynnettäviä bioaktiivisia polyfenoleja, niiden talteenottoa on tutkittu yhä enemmän markkinahinnan alentamiseksi ja perunajalostajien kannattavuuden parantamiseksi. Nykyiset uuttotutkimukset eivät kuitenkaan huomioi teknis-taloudellista kannattavuutta, tai sisällä prosessinkehitystä ja optimointia. Tässä opinnäytetyössä verrattiin modernia (ultraääni-avusteinen, UAE) ja perinteistä (kuumarefluksi, HRE) uuttomenetelmää sekä kahta esikäsittelymenetelmää (märkä, kuiva), ja optimoitiin prosessi teknis-taloudellisesta näkökulmasta. Koesuunnittelu (DoE), yhdistettynä teknis-taloudelliseen analyysiin, optimoi prosessin kannattavuuteen ja selektiivisyyteen vaikuttavat avaintekijät seuraavasti: kuivaus ja jauhatus esikäsittelyinä, kaksi HRE-vaihetta 10 minuutin viipymäajoilla, 60 °C, L/S suhde 8 ml/g, ja 50 % etanoli-vesiliuotin. Pesuvaiheen jälkeen uuttuvien polyfenolien saanto oli 96 %, sisältäen 0.73, 0.16, ja 0.76 mg/gdb klorogeenihappoa, kahvihappoa, ja α-kakoniinia, kokonaisfenolipitoisuuden ollessa 4.82 mgGAE/gdb. Tuotantokustannusarvio oli 12 €/$kg_{ekstrakti}$. Matala L/S suhde tunnistettiin merkittäväksi prosessitalouden kannalta, ja polyfenoleja, biohiiltä, ja biolannoitetta tuottava biojalostamokonsepti voi edelleen parantaa kannattavuutta. UAE osoittautui vähemmän käytännölliseksi, vaatien lisäoptimointia teknistaloudellisten indikaattoreiden ja selektiivisyyden osalta. Vaikka pilotointia ja tuotteen puhdistusprosessin kehitystä ei tutkittu, suunniteltu prosessi vaikutti teknis-taloudellisesti toteuttamiskelpoiselta mahdollistaen arvokkaiden yhdisteiden talteenoton teollisuuden perunankuorijätteestä, edistäen samalla kiertotaloutta ja vähentäen ruokajätettä.

SYMBOLS AND ABBREVIATIONS

Symbols

wt-%	Mass percent	%
vol-%	Volume percent	%
db	Dry basis	-
ppm	Parts per million	mg/kg
T	Temperature	ºC, K
L/S ratio	Liquid-to-solid ratio	ml/g

Abbreviations

AMPK	AMP-activated protein kinase
BHA	Butylated hydroxyanisole
BHT	Butylated hydroxytoluene
CA	Caffeic acid
CGA	Chlorogenic acid
EtOH	Ethanol
HPLC/DAD	High-performance liquid chromatography with diode array detector
HRE	Hot reflux extraction
IR	Infrared
LCA	Life-cycle assessment
MAE	Microwave-assisted extraction
PPE	Potato peel extract
TBHQ	Tertiary butylhydroquinone
TEA	Techno-economic analysis
UAE	Ultrasound-assisted extraction

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DECLARATIONS

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Responsibility

The author, Emil Klefström, takes full responsibility for the content of this Thesis and has reviewed and edited the content generated by the possible use of AI tools.

1 Introduction

More than one billion tons of food waste is generated annually, representing one third of total food produced, 10 % of global greenhouse gas emissions, and costing one trillion USD (European Commission, 2026; UN, 2023). Potato (*Solanum tuberosum*) is among the most widely cultivated crops, and half of it is processed by the snack food industry (Singh et al., 2011). While usually peeled before processing, potato peel waste is expected to emit 5 million tons of CO_2 equivalents in 2030 (Martínez-García et al., 2024) due to landfilling, with methane being a significant contributor to the greenhouse gas emissions.

In addition to environmental impacts and disposal costs, valuable compounds with high biological activity, primarily chlorogenic and caffeic acids, are lost (Kumari et al., 2017; Riciputi et al., 2018; Khanal et al., 2024). Those phenolic acids are already used in several sectors, although their production remains expensive. Their concentration is an order of magnitude higher in peel compared to potato pulp (Kumari et al., 2017; Wu et al., 2012), and they could provide an additional revenue for the potato processors through extraction and pyrolysis processes. The valorization of potato peel waste would also be in line with the United Nations' Sustainable Development Goal (SDG) 12.3 aimed to halve food waste by 2030. However, since phenolic compounds typically represent under 1 % of the peel mass, conventional extraction methods may remain economically infeasible.

More cost-effective and environmentally friendly methods must be developed to commercialize the extraction process (Alves et al., 2021). However, in most of the studies aimed to optimize the extraction conditions for novel methods, economic viability is neglected through high liquid-to-solid ratios, long extraction times, impractical pretreatment procedures and solvents, and consecutive or complex extraction techniques immature for industrial processes. Additionally, the existing literature tends to either optimize conditions without practical process and energy considerations or design a pilot process without optimization or economic assessment.

2 Objectives

To address the research gaps of neglecting process economics and technical feasibility during process optimization, this Thesis aims to answer three research questions: (1) Is a novel extraction technique, ultrasound-assisted extraction (UAE), technically and economically superior to a conventional method? (2) Is the added complexity of UAE justified by its enhanced extraction yield? (3) Which operating conditions provide the best balance between phenolic recovery, glycoalkaloid co-extraction, and techno-economic feasibility? The main objective is to design an industrially viable process for extracting phenolic compounds from potato peel waste.

To answer these questions, a literature review is first conducted to characterize potato peel composition, potential applications of the products, and previously studied extraction methods and operating conditions. Next, two extraction methods, together with dry and wet pretreatment options, are screened experimentally. Finally, response surface methodology (RSM) is used to optimize phenolic recovery while minimizing glycoalkaloid co-extraction. Techno-economic constraints are considered throughout the process design.

3 Potato peel characterization and valorization potential

Potato composition varies considerably because over 4000 varieties are cultivated under diverse agronomic and environmental conditions (Jimenez-Champi et al., 2023). Especially the concentration of the bioactive compounds is affected by potato variety, growing and storage conditions and periods, and environmental stresses such as light exposure, temperature, and mechanical damage (Rodríguez-Martínez et al., 2021). Most phenolic compounds are found in the peel, and it represents 10 - 40 % of the tuber (Rodríguez-Martínez et al., 2021). The share between the peel and pulp depends on the peeling technique (abrasive or steam peeling), therefore directly affecting the phenolic compound concentration in the peel. Moreover, extraction solvents and analytical methods recover phenolic compounds with varying efficiency. Potato chip and frozen French fry production

account for the majority of industrial potato consumption, together consuming up to 50 % of all potatoes produced (Singh et al., 2011; Vaduva, 2023). Potatoes are generally peeled by processors who generate revenue from the peeled potatoes, whereas the peel is often treated as a low-value by-product or waste.

3.1 Proximate composition

Proximate analyses of potato peel are collected to Table 1. All values reported are in percentages (dry basis, db), and a range of values indicate that different potato varieties were assayed.

Table 1. Proximate composition of potato peels (%, db).

Carbohydrates	Protein	Lipid	Ash	Reference
73–77	11–12	1–2	5–7	Kumari et al., 2017
67–88	6–16	0.3–2.6	2.6–5.8	Jimenez-Champi et al., 2023
87	6.5	0	5.5	Amado et al., 2014
72	10	8	9	Rowayshed et al., 2015
73	17	1	9	Rodríguez-Martínez et al., 2021
46-77	8-17	0.8-2.5	6-11	Almeida et al., 2023

The carbohydrate fraction comprises mainly starch, pectin, cellulose, hemicellulose, and sugars. Moreover, the reported lignin content varies, and in most studies, it has been neglected or included within the total carbohydrate fraction. According to some authors (Rodríguez-Martínez et al., 2021; Almeida et al., 2023), the amount of lignin varies from nearly zero to over 30 %. Similarly, the amount of pectin ranges 10 - 15 %. Starch constitutes around 50 % of the carbohydrates, and its amount increases with the peel thickness. The remaining fraction comprise cellulose, hemicellulose (Rodríguez-Martínez et al., 2021; Almeida et al., 2023) and 1.3 - 1.8 % (db) of sugars (Jimenez-Champi et al., 2023). Moreover, the peels contain approximately 1.3 % of minerals with potassium, sulfur, and phosphorus being the main constituents. The peel is also a major source of iron, magnesium, and vitamins C, B1, B6, and B9 (Rodríguez-Martínez et al., 2021). The fresh peel moisture content is 75 - 90 % across the studies.

3.2 Bioactive compounds

Potato tubers contain bioactive compounds for physiological and defensive purposes, which also explain their higher concentration in the peel compared to the pulp. The compounds can be categorized as follows: phenolic acids, anthocyanins which are mainly found in colored varieties, other flavonoids such as catechin, quercetin, and rutin complementing the peel color and flavor, non-phenolic glycoalkaloids (mainly α-chaconine and α-solanine) which possess strong antimicrobial properties and are toxic in high quantities, and carotenoids. Phenolic acids comprise chlorogenic acid (CGA; 5-*O*-caffeoylquinic acid), caffeic acid (CA), p-coumaric acid, ferulic acid, and gallic acid in measurable quantities. CGA and CA are the major compounds and cover most of the potato peels' antioxidant properties (Rodríguez-Martínez et al., 2021; Joly et al., 2021). Therefore, their concentrations are specifically considered when optimizing the extraction conditions. Of these two, chlorogenic acid is larger molecule, comprising caffeic acid and quinic acids which are attached by an ester bond. The molecules are visualized in Figure 1.

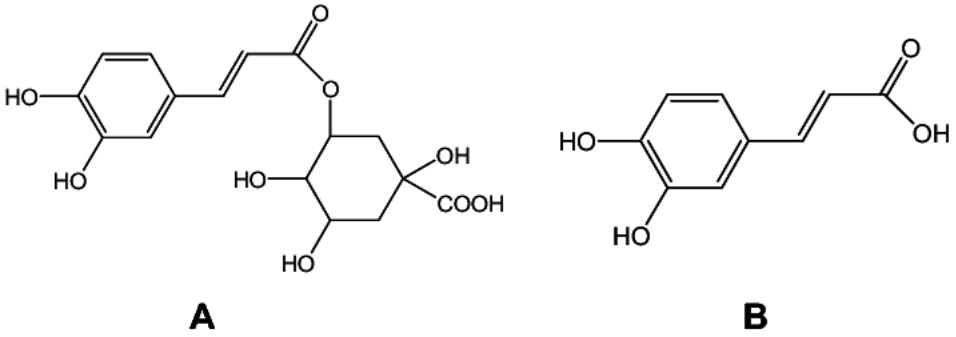

Figure 1. Chemical structures of (A) chlorogenic and (B) caffeic acid (adapted from Schieber, & Saldaña, 2009).

The bioactive compounds present in potato peel are grouped, and their quantities are shown in Table 2. Polyphenols are divided into phenolic acids (of which CGA and CA are shown) and flavonoids, which are further divided into anthocyanins and flavonols. All values are in dry basis, and a range of values indicate different potato varieties were studied.

Table 2. Concentrations of various bioactive compounds in potato peels, shown in ppm (mg/kg dry basis).

CGA, ppm	CA, ppm	Anthocyanins, ppm	Flavonols, ppm	Glycoalkaloids, ppm	Reference
435 - 6000	272-3000	30 - 1300	0 - 2040	70 - 3580	Jimenez-Champi et al., 2023
170 – 4100	160 - 3330	2 - 68	N/A	194 - 26646	Rodríguez-Martínez et al., 2021
435 - 905	272 - 697	239 - 747	273 - 740	N/A	Makori et al., 2022

While the amounts vary greatly, most potato varieties contain approximately 700 ppm of CGA and 400 ppm of CA (Jimenez-Champi et al., 2023). The other acids are in a magnitude of 100 ppm. Younger potatoes and short storage time might result in higher phenolic (Almeida et al., 2023) and lower glycoalkaloid (Khanal et al., 2024) contents. At high concentrations (>200 ppm) glycoalkaloids disrupt nervous system and cell membrane functions (Khanal et al., 2024) in most organisms.

3.3 Potential applications of potato peel fractions

The extraction process profitability can be maximized by integrating a biorefinery approach (Almeida et al., 2023), where each stream is utilized into multiple products with little waste. The following subchapters discuss applications for the streams obtained after a solid-liquid extraction process of potato peels.

3.3.1 Crude extract

Crude extract denotes the liquid part, mainly comprising polyphenols, glycoalkaloids, and sugars. Although marginally valued as such, its use have been studied in the literature. Crude extract contains multiple bioactive compounds, enabling positive synergistic effects in addition to eliminating the need for expensive purification (Almeida et al., 2023).

Synthetic antioxidants, such as BHA and BHT, have been associated with carcinogenic effects (Rowayshed et al., 2015) and environmental pollution, yet they remain widely used in processed food and cosmetic products (Cesareo et al., 2024). Potato peel extract (PPE), in turn, possesses anticarcinogenic effects. In many studies, PPE is comparable to synthetic

antioxidants (TBHQ, BHA, BHT) in preventing lipid oxidation (Amado et al., 2014; Rowayshed et al., 2015; Wang et al., 2020). One study found PPE-spiked biscuits to be inferior to controls (Rowayshed et al., 2015) while another showed PPE to even improve food color, sensory, and nutritional properties (Frontuto et al., 2019). Besides decelerating the rise in peroxide value, the phenolic mixture may be tailored for microbial prevention by adjusting its glycoalkaloid concentration. PPE has also been shown to decrease the number of harmful bacteria in intestinal and modify glucose and lipid metabolisms; therefore obstructing diabetes, obesity, and liver damage (Rodríguez-Martínez et al., 2021; Frontuto et al., 2019). In conclusion, potato peel crude extract would provide numerous benefits if it replaced synthetic antioxidants in industrial food products.

3.3.2 Phenolic acids and glycoalkaloids

A portion of the crude extract can be further purified into individual compounds. Although such post-processing is expensive requiring techniques such as membranes, precipitation, and chromatography, the products are significantly more valuable (Almeida et al., 2023).

Chlorogenic acid (CGA) has been shown to inhibit cancer proliferation and possess chemoprotective effect by regulating phase 2 detoxifying enzymes (Riciputi et al., 2018). This suggests it could be used in cancer therapy. Moreover, CGA may regulate lipid and glucose uptake through AMPK enzyme, thus enhancing insulin sensitivity, glucose tolerance, and relieving dyslipidaemia and hepatic steatosis (Riciputi et al., 2018). In other words, it holds strong anti-diabetic, anti-obesity, anti-cancer, and antihepatotoxic effects.

CGA has also been associated with neuro- and cardioprotective effects, and an ability to prevent the formation of free radicals and inflammation (Frontuto et al., 2019). Further, chlorogenic acid and its isomers (neochlorogenic acid, cryptochlorogenic acid) can be hydrolyzed into caffeic acid and quinic acid, key platform chemicals for medicine production (Sánchez et al., 2014). Likewise, glycoalkaloids are applicable for production of pharmaceutical, cosmetics, and pesticide chemicals: especially hormones, antibiotics, and fungicides (Sánchez et al., 2014; Alves et al., 2021; Jimenez-Champi et al., 2023; Almeida et al., 2023; Frontuto et al., 2019). Anti-microbial, anti-viral, anti-inflammatory, anti-allergic, and anti-cancer effects have been reported in the literature. Glycoalkaloids might also act as active ingredients in the treatment of certain skin diseases (Rodríguez-Martínez

et al., 2021). Flavonoids, in turn, have been used to treat hypercholesterolemia (Frontuto et al., 2019).

3.3.3 Fibers and starch

After extraction, the remaining solid residue can be valorized through aerobic or anaerobic fermentation, pyrolysis, or composting; producing ethanol, biogas, bio-oil and biochar, and fertilizer (Almeida et al., 2023), respectively. Currently, only anaerobic digestion, starch extraction, and composting hold the industrial-scale technology readiness (Almeida et al., 2023).

Starch granules can be separated by filtration or enzymatic treatment, and pectin precipitated with alcohol (Arrutia et al., 2020). The recovered starch may be used for fermentation or food additive, while the remaining cellulose-rich solid in pulping, nanocrystals, food additive, or bioplastics. Polysaccharides, in turn, are widely used as texture-modifying, thickening, and emulsifying agents, and they may also provide antioxidant and health-promoting effects, especially if esterified phenolic compounds remain (Rodríguez-Martínez et al., 2021). Together with food, polysaccharides reduce excess appetite and inhibit absorption of cholesterol and triglycerides (Jimenez-Champi et al., 2023). Potato peel - derived pectin seems suitable as a thickening and gelling agent particularly due to its high degree of acetylation (Rodríguez-Martínez et al., 2021), and it may also possess prebiotic effect and inhibition of pathogen colonization (Arrutia et al., 2020).

Potato protein is a widely used ingredient in vegan protein powders and meat substitutes due to its nearly complete and highly digestible amino acid profile. The remaining extraction effluents may, in turn, be utilized as fertilizer: supplying nitrogen, potassium, sulfur, phosphorus, and trace minerals for plant growth. This approach partially closes the carbon loop of potato production, approaching circular economy while also decreasing the reliance on highly polluting synthetic fertilizers (Khanal et al., 2024).

4 Novel extraction methods and their feasibility

Given the valuable compounds present in potato peel waste and their potential applications, efficient extraction strategies are essential. In this chapter, the most studied solvents, and novel extraction methods and their principles are discussed. A comparative summary of their efficiencies to extract polyphenols from potato peels is also included, and their feasibility at scale is argued.

4.1 The selection of solvent

Common solvents used to extract bioactive compounds from potato peels comprise mixtures of water with methanol, acetone, or ethanol. These solvents have lower dielectric constant than water alone, reducing the overall polarity (Paleologou et al., 2016) and thus increasing dissolution of broader range of phenolic compounds. From a practical point of view, however, methanol is toxic and unsuitable for food or cosmetics applications, and acetone is highly volatile and generally non-food grade. Ethanol, in contrast, is an inexpensive, non-toxic, and environmentally friendly solvent. While methanol outperforms ethanol in some studies (Rowayshed et al., 2015), the differences in chlorogenic and caffeic acid yield is often minor (Sánchez et al., 2014).

Amado et al. (2014) found the solvent composition to be the most significant factor affecting the phenolics profile: 71 % ethanol was optimal for phenolic acids, whereas 39 % was optimal for flavonoid recovery. Tailored ethanol concentration optimizes selectivity for the desired phenolic compounds, in this case chlorogenic acid, while minimizes the co-extraction of undesired compounds and therefore downstream processing costs (Frontuto et al., 2019).

4.2 Methods

Conventional solid-liquid extraction methods, such as maceration, Soxhlet, and hot reflux, are simple without requiring specific equipment. However, they consume considerable amount of usually volatile solvent, and the extraction duration is typically long. This results

in increased time, and solvent and energy consumptions to obtain sufficient yields. Additionally, high temperature and prolonged time might degrade the phenolics. Therefore, for economically feasible way to process potato peels, which contain approximately 1 % of phenolic compounds, several novel extraction methods have been introduced: ultrasound- (UAE), microwave- (MAE), IR-, and enzyme-assisted extraction methods as well as supercritical fluid, pulsed electric field, and pressurized liquid methods. These methods accelerate the diffusion kinetics and therefore decrease the extraction time. While UAE and MAE have been the most studied, effective, economically attractive, and frugal in solvent and heating requirements, they will be reviewed in more detail.

4.2.1 Ultrasound-assisted extraction

Ultrasonic sonotrode induces acoustic cavitation bubbles in the solvent, which grow and shrink until they collapse violently (transient cavitation). Bubble collapse generates localized high temperature and pressure zones, as well as shock waves and high velocity microjets. These mechanical effects disrupt plant cell walls and enhance the solvent penetration and mass transfer (e.g., Wang et al., 2020).

Lower ultrasonic frequencies result in more intense cavitation effect which increases yield but may also degrade the compounds easier (Kumari et al., 2017; Alves et al., 2021). Nevertheless, UAE mechanism does not rely on heat and is therefore appropriate for thermolabile compounds; preventing degradation or isomerization of phenolic acids. If necessary, UAE can be operated with a cooling jacket. The system is easy to operate, and it requires little solvent.

Although gentle, however, there are contradictory observations on phenolic stability: Kumari et al. (2017) continued the UAE for up to 900 minutes, and the yield did not decrease, although it already reached plateau at 180 minutes. In contrary, Alves et al., (2021) noticed oxidative degradation after excessive shaking time after extraction; even when sonication was not applied anymore. The sonication was noted to create free radicals which can react with the polyphenolic compounds afterwards. Besides, without rapid drying or compound isolation after the extraction, back-diffusion may occur.

4.2.2 Microwave-assisted extraction

Microwaves penetrate cell matrix and vaporize water, increasing intracellular vapor pressure (volumetric heating) and rupturing cell walls. Instead of thermal conductivity, the heating occurs through ionic conduction and dipole rotation (Wu et al., 2012); dielectric compounds, such as water, absorb microwaves and rotate along the oscillating electromagnetic wave. Therefore, the heating power and extraction time can be significantly reduced compared to conventional extraction methods.

The interaction between biomass and microwaves depends on the used frequency, dielectric constant (the material's ability to become polarized and absorb microwaves), and loss factor (ability to dissipate the microwave energy into heat) (Arrutia et al., 2020). These factors determine the microwave penetration, albeit being only a few centimeters or less at the standard 2.45 GHz (Sepelevs et al., 2020; Arrutia et al., 2020). Lower frequency improves the penetration depth. Moreover, it is important to note that increasing temperature increases the dielectric constant and loss factors, causing decrease in the microwave penetration depth (Arrutia et al., 2020).

Besides the low penetration depth, the use of lower frequency and a continuous plug-flow reactor seems to largely eliminate the limitation. Overall, extraction times can be reduced to few minutes or seconds, enabling low energy consumption and high throughput. Moreover, relatively low microwave power seems to be sufficient (Sepelevs et al., 2020). Although high temperatures above water boiling point are generally used, the short duration might prevent phenolic degradation. However, certain scale-up constraints remain: microwave magnetron lifetime is typically under 8000 h, requiring replacement at least yearly, and MAE system requires ATEX-rated equipment. Therefore, the higher extraction yield or lower energy consumption must justify the increased capital and maintenance costs.

4.3 Comparison of the method efficiencies

Table 3 collects studies on polyphenol extraction from potato peels. The time is chosen at which plateau or maximum yield is achieved. A yield from a comparative conventional extraction is shown in parenthesis if it was conducted in the study. UAE frequency varied between 23 and 42 kHz, and in all MAE experiments 2.45 GHz was used. In UAE studies

the power varied between 100 and 190 W, in MAE between 50 and 300 W. The power level had little impact on the yields, and it is difficult to compare since different sample amounts were used in the studies. In almost all studies the samples were freeze-dried and then milled to < 0.2 mm. The yield is expressed as total phenolic content (TPC, Folin-Ciocalteu method) and CGA concentration (HPLC). TPC unit is milligrams of gallic acid equivalents per gram dry weight of potato peel (mgGAE/gdb), and CGA in milligrams per gram dry weight of potato peel (mg/gdb).

Table 3. Optimized extraction conditions and polyphenol yields from potato peels.

Method	Solvent /solid, ml/g	Solvent & concentration	T, °C	Time, min	TPC, mgGAE/g db	CGA, mg/gdb	Reference
UAE, bath, m=0.12	81	Glycerol, 83 %	80	30	8.71	N/A	Paleologou et al., 2016
UAE, bath	84	Ethanol, 59 %	77	30	9.11	N/A	Paleologou et al., 2016
UAE, bath, (LR variety), m=1 g	10	Methanol, 80 %	30-45	180	7.67 (3.28)	0.267	Kumari et al., 2017
UAE, bath, (LC variety), m=1g	10	Methanol, 80 %	30-45	180	3.80 (2.17)	0.00598	Kumari et al., 2017
UAE, bath, (m=3g)	10	Ethanol, 55 %	35	35	6.5 (3.41)	1.3-4.1	Riciputi et al., 2018
UAE, bath, m=2g	20	Methanol, 86 %	22	28	9.95 (7.02)	N/A	Moradi et al., 2024
UAE, bath m=0.25g	20	Methanol, 50 %	25	30	9.09	N/A	Wang et al., 2020
UAE, probe m=0.25g	40	Methanol, 50 %	25	30	9.3 (6.26)	N/A	Wang et al., 2020
UAE, probe m=0.25g	40	Methanol, 50 %	25	30	9.3 (5.86);	N/A	Xu et al., 2022
UAE, m=10g	20	Ethanol, 70 %	50	N/A	1.57 – 3.6	N/A	Makori et al., 2022
UAE, m=30g, wet	2.5	Water, ethanol, acetic acid (46:51:3)	20	50	N/A	0.4	Sánchez et al., 2014
MAE, m=2g	20	Methanol, 67 %	N/A	15	3.94	1.35	Singh et al., 2011
MAE, (m=0.5g)	40	Ethanol, 60 %	80	2	10.3 (9.6)	4.71	Wu et al., 2012
MAE, m=25g wet	10	Ethanol, 60 %	79	10	3.2	N/A	Sepelevs et al., 2020
MAE, m=0.25	20	DMF	100	120	N/A	0.53	Gupta et al., 2024
IR-assisted, m= 1g	10	Distilled water	80	10	3.5	0.02	Helmi et al., 2024
Maceration	50	Ethanol, 29 %	23	50	10.31	0.25	Shaheen et al., 2021
Maceration	25	Ethanol, 50 %	20	60	3.5	0.29	Almeida et al., 2024
Maceration, wet	25	Ethanol, 60 %	20	60	1.2	N/A	Almeida et al., 2024
Hot ethanol	10	Ethanol, 80 %	75	30	3.83	0.79	Joly et al., 2021
Hot ethanol	20	Ethanol, 71 %	90	34	10.3	1.55	Amado et al., 2014
Hot ethanol, wet	20	Ethanol, 54 %	50	233	5.9	1.7	Frontuto et al., 2019
Maceration	10	Ethanol, 100 %	20	720	0.76	0.11	Rowayshed et al., 2015

TPC in potato peel extracts varies significantly between studies (Table 3). Conventional methods achieved 0.8 – 10.3 mgGAE/gdb, while UAE and MAE showed 1.6 – 9.9 and 3.2 – 10.3, respectively. Although widely used due to its simplicity, speed, and low cost, Folin-Ciocalteu assay measures reducing capacity rather than the phenolics directly (Pérez et al., 2023). Consequently, TPC values may be overestimated due to interfering compounds such as ascorbic acid, reducing sugars, and Maillard reaction products; especially in high temperature extractions. Additionally, some studies do not explicitly state whether the unit refers to dry weight or extract. Conversely, since HPLC may underestimate the phenolic concentration due to undetected compounds, the actual phenolic concentration is expected to locate between the HPLC and TPC estimates. Despite these limitations, however, TPC has been shown to correlate with chromatographically determined phenolic concentration (Amado et al., 2014; Rodríguez-Martínez et al., 2021), and it is used as the primary response variable in the experimental part of this work. To verify this assumption and prevent flawed conclusions, phenolic profiles of the best extraction conditions will also be analyzed by HPLC.

While chlorogenic acid (CGA) is usually the dominating phenolic acid in potato peels, several studies have measured higher caffeic acid (CA) contents by HPLC. CGA can be hydrolyzed into CA during too severe extraction conditions, which may partly explain the results. For example, Kumari et al. (2017) and Rowayshed et al. (2015) measured 0.12 and 0.21 mg/g CA but only 0.006 and 0.11 mg/g CGA, while Almeida et al. (2024) and Helmi et al. (2024) found 0.29 and 0.02 mg/g CGA whereas total phenolic acids reached 6.45 and 0.17 mg/g, respectively. In those studies, the extraction was performed either at moderate to high temperatures or for extended duration.

Regarding process parameters, lower ultrasonic frequency improves the extraction yield in UAE and temperature does not show any trend, whereas in conventional extraction, temperature appears to be a significant factor (Table 3). Across all the studies, potato variety and solid-to-solvent ratio consistently affect phenolic recovery, while superfluous time was unfavorable. Most studies focused on single extraction technique, and when conventional was included as a reference it often showed one-third to two-fold lower yields despite significantly longer extraction times. However, the reference extraction was often conducted at room temperature (maceration) and at higher temperatures the yield was closer to those with MAE and UAE. Wu et al. (2012) used hot reflux extraction (HRE) as a reference and

the yield was only 6 % lower compared to MAE. Kumari et al. (2017) achieved over 2 times higher TPC with UAE compared to maceration, but HPLC revealed that CGA concentration did not differ significantly. Across the studies, comparison between dry and wet (fresh) raw material remains scarce, despite its relevance for industrial processing. Therefore, this work aims to compare dry and wet raw material, and UAE is compared to HRE rather than maceration.

4.4 Process feasibility

Optimizing the extraction conditions for pure yield is deficient due to impractically high temperatures, liquid-to-solid ratios, and long extraction times. Techno-economic analysis (TEA) must be conducted along with the process optimization, since economic viability is highly affected by the operating conditions, pretreatment, and unit operations selected. This and the following subchapter discuss constraints on drying pretreatment, energy consumption, and downstream processing.

In most studies, the peel mass had been freeze-dried and blended into powder prior to extraction. Such a pretreatment is not feasible at scale due to the significant energy consumption of freeze-drying. While freeze-drying seems to release bioactive compounds from cell matrix compared to conventional freezing which reduces their amount (Martínez-García et al., 2024), many literature results can be expected to be optimistic and not reflect to the industrial optima. Moreover, since phenolic compounds begin to degrade above 100 °C (Grzelczyk et al., 2022; Gupta et al., 2024; Helmi et al., 2024), the drying process, if applied, must be gentle. The losses have been shown to increase with increasing drying temperature or time (Joly et al., 2021).

Almeida et al. (2024) concluded that drying and milling would improve economic viability due to reduced solvent requirement and enhanced yield. However, the reference scenario without drying also excluded milling. Since extraction yield is strongly affected by particle size, the comparison does not isolate the effect of drying. Up to 150 % increase in yield can be obtained with particle size reduction from 2 mm to 0.75 mm due to increased interface-area-to-mass ratio and shorter diffusion path (Ćujić et al., 2016; Wang et al., 2020). Additionally, only conventional extractions were carried out by Almeida et al. (2024), and

solvent recovery was based on evaporation which over-estimates the operating costs due to its high energy demand. Also, the conclusion was based on TPC instead of HPLC assay.

Finally, it must be noted that while smaller particle size enhances yield, it requires more milling energy, and unnecessary small particle size might complicate the solid liquid separation after the extraction and blind filters. Therefore, optimal particle size is expected to occur. For scale-up reasons, higher particle size will be retained in this work, especially because wet material cannot achieve powder-like particle size. The aim is to isolate the drying effect.

4.5 Downstream processing

Due to decreased polarity compared to pure water, 50-60 % ethanol solvent reduces solubility of large polysaccharides, while low molecular-weight compounds remain soluble (Sepelevs et al., 2020). Consequently, the extract is expected to be enriched in phenolic compounds and glycoalkaloids, along with sugars, short oligomers, organic acids, amino acids, vitamins, and minerals (Table 1).

Further purification for polyphenols could be achieved by ultrafiltration followed by nanofiltration. Glycoalkaloids are considerably larger molecules than phenolic acids (CGA: 354 g/mol; α-chaconine: 852 g/mol) and are therefore expected to retain in ultrafiltration membrane. Nanofiltration could then separate phenolics from smaller solutes such as glucose (180 g/mol) and minerals. However, the molar masses are within the same magnitude, and phenolic acids are known to adsorb onto polymeric membranes (Filippou et al., 2022), causing unstable separation, membrane fouling, and product losses. Therefore, membrane selection and operating conditions must be carefully optimized.

In addition to purification, membrane filtration enables solvent recovery. Compared to thermal evaporation, membrane separation demands two to three orders of magnitude less energy (Larsen, 2022), improving process viability. Although membrane fouling and concentration polarization reduces flux, cross-flow operation, turbulence promoters, and prefiltration immediately after the extraction may mitigate these effects. Simultaneously, back-adsorption of the polyphenols back into the solid matrix is minimized.

At high temperatures, especially after MAE or in wet processing, starch gelatinization may increase extract viscosity and complicate downstream processing, especially at liquid-to-solid ratios below 10 ml/g. Gelatinized starch might also adsorb the polyphenols. Therefore, Arrutia et al. (2020) proposed recovering the starch prior to extraction using a 150 µm sieve. Such pre-treatment might also reduce the amount of soluble sugars in the extract, preventing the overestimation of TPC. Optimized extraction time, temperature, pH, and ethanol concentration may also limit the back-adsorption. Moreover, product drying under vacuum or inert gas, low temperature, and acidic conditions improve the polyphenol stability during storage.

5 Materials and methods

This chapter describes materials, procedures, and methods used to evaluate the polyphenol extraction performance. Figure 2 shows the simplified process flow that was used in the experimental part of this work.

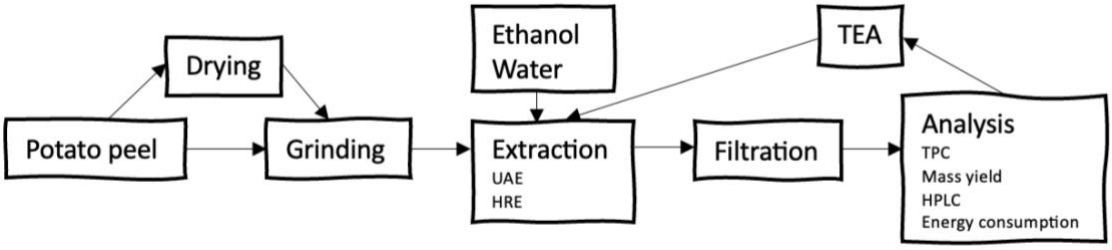

Figure 2. A simplified block diagram of the experimental procedure.

5.1 Chemicals and reagents

The chemicals used in this study included ethanol (CAS 64-17-5), methanol (CAS 67-56-1), phosphoric acid (orthophosphoric acid, CAS 7664-38-2), sodium carbonate (Na_2CO_3, CAS 497-19-8), Folin–Ciocalteu reagent (CAS 52503-77-8), α-chaconine (CAS 20562-03-2), gallic acid (3,4,5-trihydroxybenzoic acid, CAS 149-91-7), chlorogenic acid ((1S,3R,4R,5R)-3-{[(2E)-3-(3,4-dihydroxyphenyl)prop-2-enoyl]oxy}-1,4,5-trihydroxycyclohexane-1-carboxylic acid, CAS 327-97-9), and caffeic acid ((2E)-3-(3,4-dihydroxyphenyl)prop-2-

enoic acid, CAS 331-39-5). They were bought from Merck (Darmstadt, Germany) and were of analytical or HPLC grade.

5.2 Samples

The potatoes (Folva variety, Finland) were bought from a local supermarket (Lappeenranta, Finland) in two batches in March and June 2026. Within 2 days, they were manually washed, peeled, and ground using a blender. A portion of the peels were dried overnight at 60 °C prior to blending. Particle size was maintained at 0.71 mm by sieving. After grinding, the peels were stored in a refrigerator in airtight tubes for a maximum of 24 h. The moisture content of the peels was determined by weight loss on overnight at 105 °C.

5.3 Experimental design

Two experimental designs were conducted: (1) Screening to select the best pretreatment (wet, dry) and extraction method (ultrasound-assisted extraction (UAE), heat reflux extraction (HRE)), and (2) Box-Behnken and Full Factorial designs to optimize the conditions obtained from the screening. The process was optimized under techno-economic constraints, including measurements or estimations of milling, drying, extraction, solid-liquid separation, and solvent recovery energies. A mixture of water and ethanol was used in all experiments as a solvent.

5.3.1 Screening

The aim of the initial screening was to isolate the effects of extraction method and raw material. Compared to dried raw material, wet peels require two times more ethanol for processability at an equivalent liquid-to-solid ratio and are expected to obtain lower polyphenol yield. Therefore, the screening also evaluates whether avoiding energy-intensive drying justifies the higher solvent demand and lower extraction yield. The fixed factors were selected based on the results from previous studies (Table 3) and are shown in Table 4. The independent factors were extraction method (UAE, HRE), temperature (50 °C, 70 °C) and drying (wet, dry). A total of 8 experiments were conducted using Full Factorial design, and

they were duplicated at 70 °C. In addition, kinetic experiments were conducted by taking samples with time. The experiments were run up to 30 minutes, except for separate HRE dry and wet experiments which were extended to 90 min to confirm plateau.

Table 4. The fixed independent factors used in the screening experiments.

Method	Ethanol concentration, %	Liquid-to-solid ratio, ml/g db	Duration, min
UAE	50	15	30
HRE	50	15	30

5.3.2 Optimization

After selecting the extraction method and pretreatment, the process was further optimized using Box-Behnken and Full Factorial designs. The independent factors in Box-Behnken were temperature (40-80 °C), ethanol concentration (30-70 %), and liquid-to-solid (L/S) ratio (5-20 ml/g); each in three levels (min, max, center). All experiments were run for 30 minutes and in a randomized order. Response surface methodology (RSM) was applied to find the relationships between the factors. RSM also revealed optimal conditions that minimizes production costs (section 5.6), which was the response variable. It was calculated using total phenolic content (section 5.5.1).

The Box-Behnken design consisted of 15 experiments including three replicates at central points. A quadratic model (equation 1) was fitted to predict the response variables as a function of the independent factors:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad (1)$$

where Y is the predicted response (production cost, [€/$kg_{extract}$]); X_1, X_2, and X_3 are independent factors; β_0, β_i, β_{ii}, and β_{ij} are the intercept, and regression coefficients of the linear, quadratic, and interaction terms of the model, respectively. The design matrix is shown in Table 5.

Table 5. Box-Behnken design matrix before coding and randomization.

Run	Temp, °C	EtOH, vol-%	L/S, ml/g
1	40	30	12.5
2	40	70	12.5
3	40	50	5
4	40	50	20
5	80	30	12.5
6	80	70	12.5
7	80	50	5
8	80	50	20
9	60	30	5
10	60	30	20
11	60	70	5
12	60	70	20
13	60	50	12.5
14	60	50	12.5
15	60	50	12.5

Full Factorial design was used to further optimize ethanol concentration (40-60 %) and L/S ratio (8-12 ml/g) at 3 levels. The design comprised nine 30-min experiments, and similar quadratic model as in equation (1) was fitted except only 2 factors were used (6 coefficients instead of 10). The design matrix is shown in Table 6.

Table 6. Full Factorial design matrix before coding and randomization.

Run	EtOH vol-%	L/S, ml/g
1	40	8
2	50	8
3	60	8
4	40	10
5	50	10
6	60	10
7	40	12
8	50	12
9	60	12

5.4 Extraction procedures

Conventional extraction (heat reflux extraction, HRE) was conducted with a jacketed glass vessel connected to a hot water bath, air-cooled condenser, and magnetic stirrer. UAE comprised a jacketed vessel, transducer (UW2200, Bandelin, Germany), sonotrode (Sonopuls VS70T, Bandelin, Germany) and ultrasonic generator (Sonopuls, Bandelin, Germany). The frequency was 20 kHz, and maximum power 200 W. 15 % amplitude and pulse mode (10s on, 5s off) were used. Parafilm was applied around the probe to prevent solvent evaporation. The mixing was sufficient without magnetic stirrer.

Briefly, 1 g (dry) or 6.7 g (wet, corresponding to 1 g dry basis) of grinded peels were mixed with solvent in the vessel. Immediately after extraction, the mixture was decanted into sealed tube, and it was centrifuged for 10 minutes at 2000g. Alternatively, Buchner filtration (Whatman 2-3 µm cellulose filter paper) was performed before centrifugation to measure filterability and the amount of recovered liquid. The supernatant was then pipetted to another tube, TPC was measured using the Folin-Ciocalteu method (section 5.5.1), and mass yield was determined according to equation (2). The production costs were calculated (section 5.6), and the extract from each best method condition was analyzed by HPLC (section 5.5.2) to obtain actual phenolic content and phenolic profile. The experiments were triplicated for statistical confidence. For reference, two experiments (dry, wet) were conducted by only shaking the peels and solvent in a sealed falcon tube for 3 minutes at room temperature.

5.5 Analytical methods

Folin-Ciocalteu method and HPLC were used to measure total phenolic content (TPC), and individual polyphenol and glycoalkaloid concentrations, respectively. Moreover, the extract gravimetric yield was defined by overnight drying at 105 °C according to equation (2). The prewarmed watch glasses and later samples were cooled in a desiccator before weighting.

$$Y_{mass} = \frac{m_{dried_extract}}{m_{original}} \frac{V_{solvent}}{V_{aliquot}} * 100\ \% \quad (2)$$

where $m_{dried_extract}$ is dry mass obtained from the aliquot [g]; $m_{original}$ is dry-basis mass of potato peels used in the extraction [g]; $V_{solvent}$ is total extract volume [ml]; $V_{aliquot}$ is the volume of extract used for drying [ml]. The equation gives the percentage of sample mass (db) that was shifted into the liquid phase during the extraction.

5.5.1 TPC assay

Total phenolic content (TPC) was quantified using Folin-Ciocalteu method, as reported by Wang et al. (2020) with modifications. Briefly, 50 µl of 4x-diluted sample or standard solution was transferred into a glass tube with an electronic pipette. 1250 µl of 10x-diluted Folin-Ciocalteu reagent was added and vortexed. After 3 minutes, 1000 µl of 7.5 % Na_2CO_3 solution was added, vortexed, and incubated at 45 °C for 15 minutes. The liquids were sequentially poured into a glass cuvette, and absorbance was read at 765 nm by UV/Vis spectrophotometer (V670, Jasco, Japan). Pure water was used as a blank. Samples were prepared in triplicates. Calibration curve (gallic acid standard) was prepared at 0 - 600 mg/l, and the coefficient of determination was over 0.999. The measured TPC was expressed as milligrams of gallic acid equivalents per gram dry weight of potato peel (mgGAE/gdb); by multiplying the calibration curve -based value by solvent volume and dividing by sample mass (dry basis) used in the extraction.

5.5.2 Identification of phenolic acids and glycoalkaloids

HPLC/DAD was used to determine chlorogenic acid, caffeic acid, gallic acid, and α-chaconine. Standards were prepared at 5-400 mg/l. HP 1200 (Agilent Technologies, Palo Alto, CA, USA) was used, and the column (Kinetex 2.6u C18 100A, Phenomenex, USA) and conditions were selected as in Sánchez et al. (2014). The compounds were identified based on retention time, at 280 nm and 202 nm wavelengths. Phenolic acids and α-chaconine were determined with two separate runs.

5.6 Techno-economic analysis

To obtain a coarse estimation of the production costs, a light gate-to-gate techno-economic analysis (TEA) was conducted using a Python script. Briefly, the measured energy consumptions and estimations for solvent loss and electricity price were used to calculate the total energy and solvent costs per unit (1 kg raw material db). The cost was divided by the polyphenol yield to obtain production cost in [€/$kg_{extract}$]. Drying cost was estimated to be 0.9 kWh/kg_{wet}, which is an average energy consumption of commercial dryers (Havlík et al., 2020). 97 % of solvent (ethanol) was expected to be recovered in the process. Grinding energy was measured, and extraction energy was estimated based on solvent heat capacity, and efficiency of 40 %. Solvent cost was estimated to be 0.45 €/kg (ethanol/water 50/50), and peel cost 0.05 €/kg. The lowest practical liquid-to-solid ratios were used (15 ml/g for wet, 5 ml/g for dry). For simplicity, the estimations did not include fixed overhead costs (FOC). Further, the production costs were used to calculate profit rate as follows:

$$P = \frac{SP - PC}{t_s} * m_E \quad (3)$$

where: P = profit rate [€/h/kg_{peel_db}]; SP = estimated selling price (40 €/$kg_{extract}$); PC = production costs based on the calculations above [€/$kg_{extract}$]; t_s = residence time in the process (0.5 h); m_E = amount of extract obtained from 1 kg of peel [$kg_{extract}$/kg_{peel_db}].

5.7 Statistical analysis

One-way analysis of variance (ANOVA) was used to determine statistical significance of the Box-Behnken and Full Factorial model coefficients (quadratic). Statistical significance is based on $p < 0.05$. Linear ordinary least squares (OLS) model was used to fit the coefficients and predict the responses. R^2, Q^2, F value, and p value were used to verify the models' finesses.

The analyses were performed using a Python program. Independent variables were scaled to dimensionless values (−1 to +1) to improve numerical stability and allow comparison of the coefficients. Data is reported as mean value ± standard deviation of triplicates or duplicates, depending on the experiment.

6 Results

This chapter presents the experimental results of the pretreatment and method screening, and process optimization. The extraction performance was evaluated using total phenolic content (TPC, Folin-Ciocalteu), mass yield, phenolic profile (HPLC), and techno-economic indicators. The primary response used to optimize the process was profit rate [€/h/kg_{peel_db}].

6.1 Raw material characterization

The peel moisture content was 84.7 %, and it is consistent with literature (75-90 %, Table 1). Wet peels required liquid-to-solid ratio of 10-15 ml/g for processability, depending on the ethanol concentration, and the extraction vessel had to be shaken periodically for homogenous mixing. For dry peels, liquid-to-solid ratio of 5-8 ml/g was sufficient to maintain mixing; however, most solvent adsorbed into the dried peel mass during the extraction and little solvent could be recovered. With 8 ml/g of solvent, 3-5 ml/g was recovered after Buchner filtration. The recovery increased with ethanol concentration. It is supposed that the dry peel adsorbed water but not ethanol, and therefore a solvent with a higher ethanol content also recovered better. In all cases, the solid settled within a few minutes. Starch gelling was not observed during the extractions.

6.2 Screening

The screening experiments compared ultrasound-assisted extraction (UAE) and conventional hot reflux extraction (HRE). Both methods employed dry and wet processing, and two temperatures. Total phenolic content (TPC, Folin-Ciocalteu), mass yield, and production costs were calculated for each condition.

6.2.1 Total phenolic content and mass yield

TPC and mass yield (equation 2) are shown in Figure 3. TPC is reported as mean value ± standard deviation (n=3).

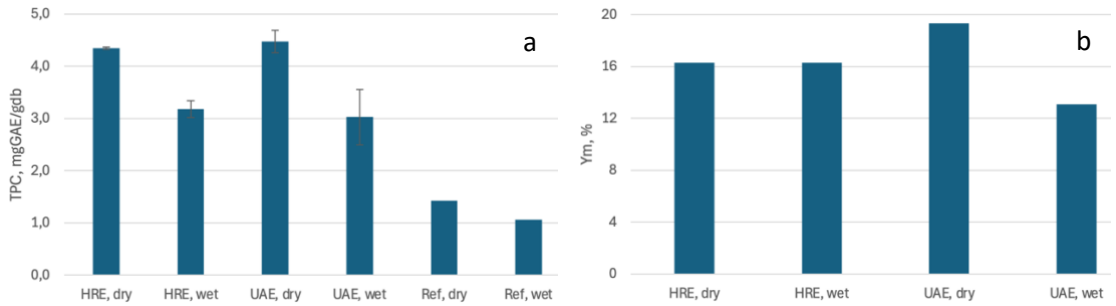

Figure 3. Total phenolic contents (a) and mass yields (b) in the screening studies.

Literature (Table 3) reports TPC between 2 and 10 mgGAE/gdb. The yields obtained in this work (3 – 5 mgGAE/gdb) fall within that range. Nevertheless, the yield difference between HRE and UAE was not significant ($p = 0.48$, $n = 3$), and both methods performed better compared to the references (short shaking without heat). Likewise, with both methods, dry peel extraction achieved approximately 1.36 times higher yield compared to wet, and it was statistically significant for HRE ($p < 0.05$) but not for UAE ($p = 0.07$) due to high variation between UAE wet experiments. HRE achieved more consistent dry and wet yields, and the wet process achieved higher TPC compared to wet UAE. Wet peel grinding already disrupted the cell matrix physically, which may explain the results. Moreover, UAE might have extracted polyphenol aggregates which settled during centrifugation and therefore they were not recovered.

TPC and mass yield seems proportional, which is expected due to higher concentration of polyphenols, ascorbic acid, and reducing sugars inflating the TPC value. According to Figure 3, 12 – 20 % of the raw material (dry basis) was recovered in the extract. While polyphenols account under 1 % of dry peel (Table 2), significant amounts of sugars, short oligomers, organic acids, amino acids, vitamins, and minerals had co-extracted. Further, the gravimetric yield was measured from centrifuged extract (HRE, dry) supernatant, and it was 12 %. Before centrifugation the yield was 16 % in HRE dry, revealing that most of the solid is dissolved and not settleable. While gravimetric yield measurement of UAE extract before and after centrifugation remained untested, the assumption of higher aggregate extraction during UAE could not be verified.

6.2.2 Energy consumption

Energy consumptions between the extraction methods could not be directly compared, since scale-up affects the methods differently: UAE typically becomes more energy-intensive at scale (Tamminen et al., 2022), while HRE benefits from lower relative heating losses in jacketed reactors. Therefore, TEA was only calculated for wet and dry peels generally, using HRE yields. According to the established TEA (Figure 4), wet and dry extractions showed productions costs of 19.9 and 18.5 €/$kg_{extract}$, respectively. Although wet peel extraction was not preceded by energy-intensive drying, its grinding and extraction costs were 2 times higher due to higher wet peel and solvent masses. Moreover, in wet processing, 7.8 ml of ethanol and 2.2 ml water were added to 6.7 g of wet peels (which contains 5.6 ml water) to obtain 50 % ethanol concentration dry basis. In dry process, 2.5 ml of both ethanol and water was adequate, although not optimal, for mixing. Therefore, wet process consumed 3 times more ethanol in the experiment. With an S/L ratio of 8 ml/g, the dry process would still consume 2 times less ethanol.

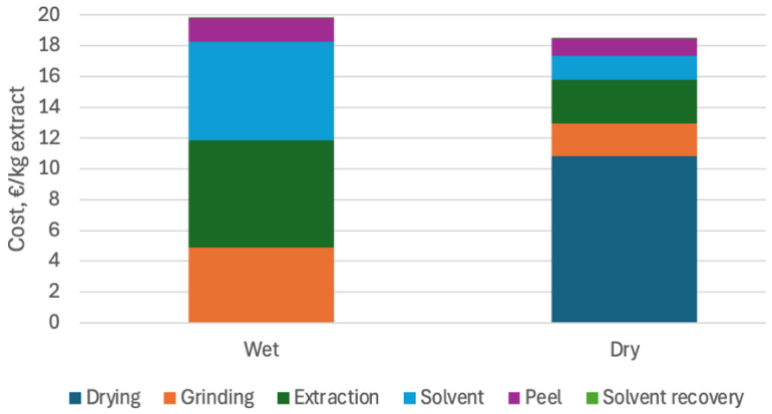

Figure 4. Itemized production costs for HRE extractions.

With dry peels, drying cost was clearly the dominating factor affecting production cost. In both cases, solvent recovery cost using membrane (nanofiltration) separation was negligible, accounting under 0.1 % of the total costs. Solvent distillation would consume considerably more energy, although membrane fouling issue is avoided in that case. With thermal solvent recovery, the gap between wet and dry process viability would further inflate due to more solvent to be evaporated in the wet process. Moreover, dry peel process costs could be significantly reduced by using waste heat for drying, while in wet process lower ethanol concentration, solvent recovery optimization, and shorter residence time may reduce costs.

Decreasing liquid-to-solid (L/S) ratio in wet process was noticed to produce too thick slurry for extraction. Finer milling with a wet hammer mill, for example, may enable lower L/S ratio; however, it also increases grinding energy and may increase starch gelling.

6.2.3 Kinetic experiments

Several kinetic comparison experiments were performed to better understand the system kinetics. Figure 5 shows comparison of each condition. 70 °C experiments are shown in red, 50 °C experiments in blue, UAE with square markers, HRE with circle markers, dry experiments with filled markers and wet experiments without fill. The points are connected with thin lines for readability.

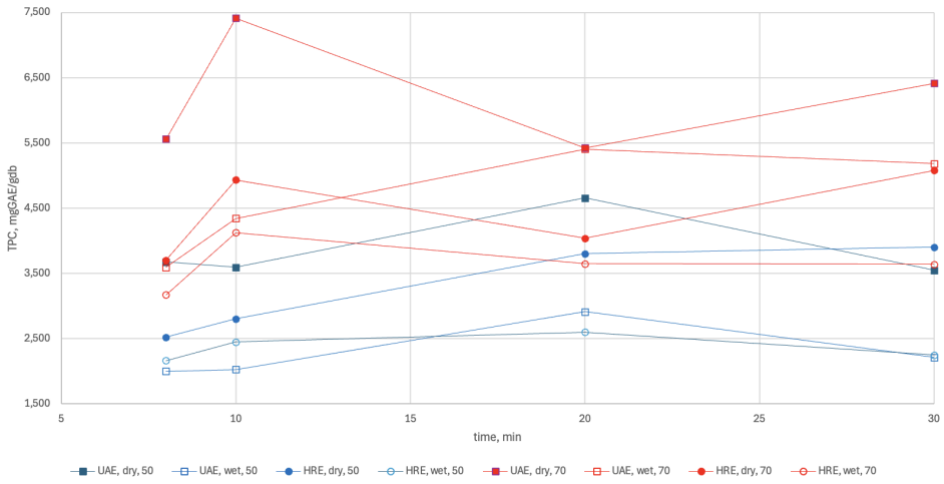

Figure 5. Kinetic experiments of each method configurations.

Although severe noise and possible back-adsorption, the results conclude that the yield increases with high temperature, UAE, and dry process. For confidence and to change less variables at once, the experiments were duplicated one variable at a time. Figures 6, 7, and 8 show kinetic comparisons of HRE at different temperatures, HRE with dry and wet pretreatment, and HRE dry compared to UAE dry and wet, respectively. L/S ratio of 15 ml/g was used in each experiment.

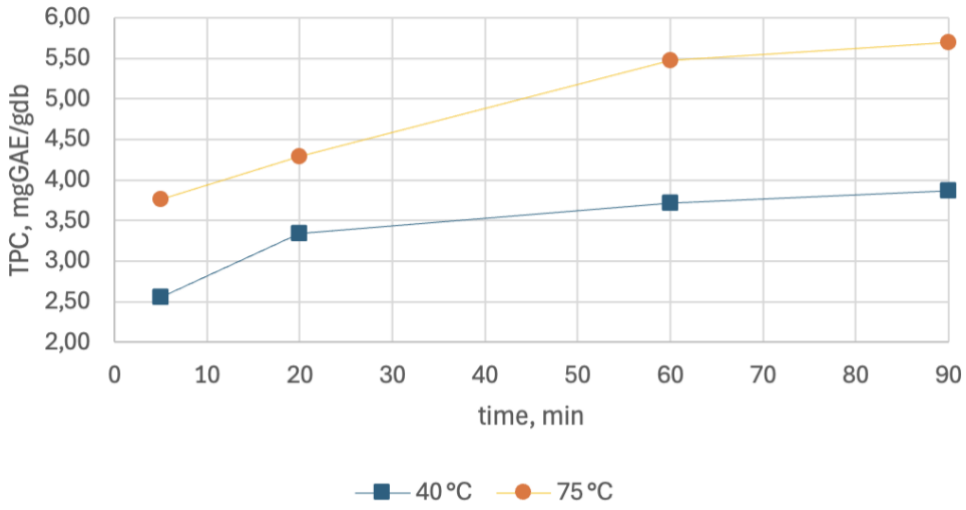

Figure 6. Kinetic comparison of HRE at 40 and 75 °C.

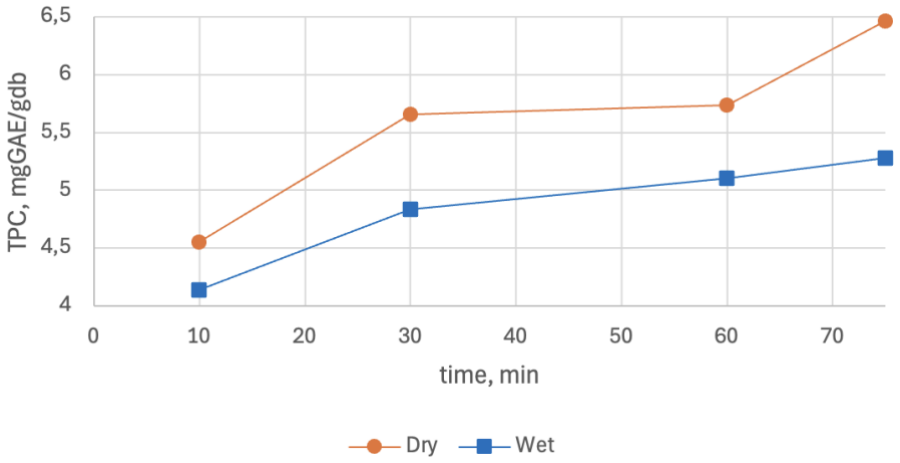

Figure 7. Kinetic comparison of dry and wet HRE.

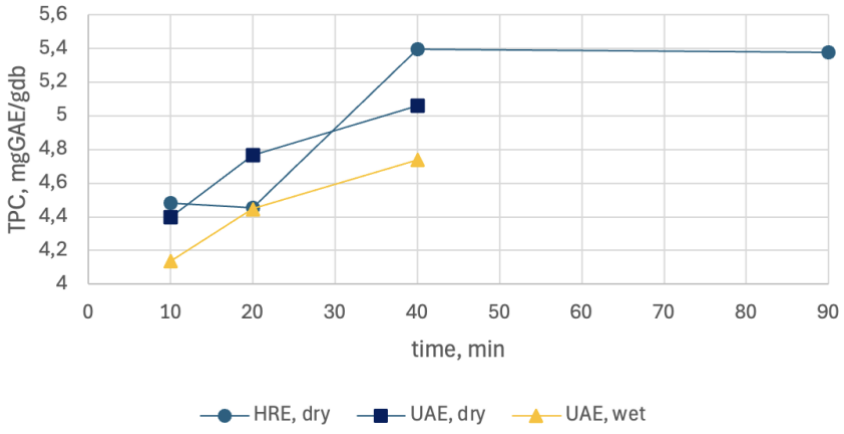

Figure 8. Kinetic comparison of HRE dry and UAE dry and wet.

Figures 6 and 8 imply HRE dry reaches plateau at between 40 and 60 minutes, while in Figure 7 the yield seems to still increase after 60 min. Overall, the kinetics seems to be slightly faster at high temperature (Fig. 6) and in dry process (Fig. 7 and 8).

6.2.4 Selection of conditions for further study

HRE was not significantly inferior to UAE. Moreover, dry process achieved lower production cost and more consistent yields compared to wet. Therefore, dry HRE was chosen for further study due to its simplicity; at scale, a jacketed CSTR with high technology readiness level would suffice as the extractor compared to a tubular sonoreactor that must be specially designed if UAE is chosen. Steam-heated CSTR reactors are highly energy-efficient. The downside in conventional extraction is longer extraction time; nevertheless, larger CSTR volume decreases residence time. Further, dry process enabling lower liquid-to-solid ratio requires lower ethanol consumption, simpler downstream processing, lower volume to be processed, higher product concentration, and therefore smaller equipment. That would also reduce fixed capital costs.

Filterability and settleability were sufficient in each experiment, and therefore, they were not used as criteria. However, it is important to consider that dried sample containing fine dust may blind filter press at industrial scale. Nevertheless, according to Buchner filtration experiments, the dry filtration speed was significantly faster compared to wet sample. It is hypothesized that wet peel released pectin and starch into the filtrate, creating a gel layer onto the filtration paper; although, it did not increase the solvent viscosity, and therefore the gel could not be visually observed during extraction. In contrast, no filter blinding from fine particles was observed during filtration of the dry-process extract, likely due to low solid load on the paper. The paper was replaced between the filtration experiments ($V_{recovered_extract}$ = 5-10 ml). To assess the filter blinding from fine particles, one experiment was conducted with higher dry-process extract volume (V = 50 ml). The filtration rate decreased significantly during the filtration and eventually the paper clogged.

While in dry process the drying clearly dominates the production cost, drying energy may be reduced by mechanically dewatering the wet peel before thermal drying. Furthermore, the peel mass could not be properly milled without drying. The wet peels could only be scissored into approximately 5x1 mm sticks. When the scissored sticks were compared to

scissored sticks which were also ruptured with a mortar and pestle simulating industrial rod mill, the yields were identical. In other words, cell wall disruption did not enhance the yield in the case of wet peel. On the contrary, dried and finely milled peel enabled the highest yield while dry coarse particle size yield fell between the dried and finely milled, and wet milled peel yields. This reveals the yield is affected by both the particle size, which increases surface area, and the drying process, which increases porosity. The drying effect will be further discussed in the section 6.3.7.

6.3 Optimization

The most promising extraction procedure, dry HRE, was further optimized using 3-factor Box Behnken and 2-factor Full Factorial designs. The factors were temperature (only in Box-Behnken), ethanol concentration, and liquid-to-solid ratio. 3 levels were used in both designs. Before the design experiments, a kinetic study was conducted by taking samples over time at two separate conditions (high and low severity) to determine an appropriate extraction time. The time at which 80 % of the plateau yield was reached (t_{80}) was selected, which was approximately 30 min.

6.3.1 Box-Behnken RSM

The response surfaces of the Box-Behnken design are shown in Figure 9. The response is profit rate (section 5.6). Model parameters are shown in Table 7 and coefficients in Table 8.

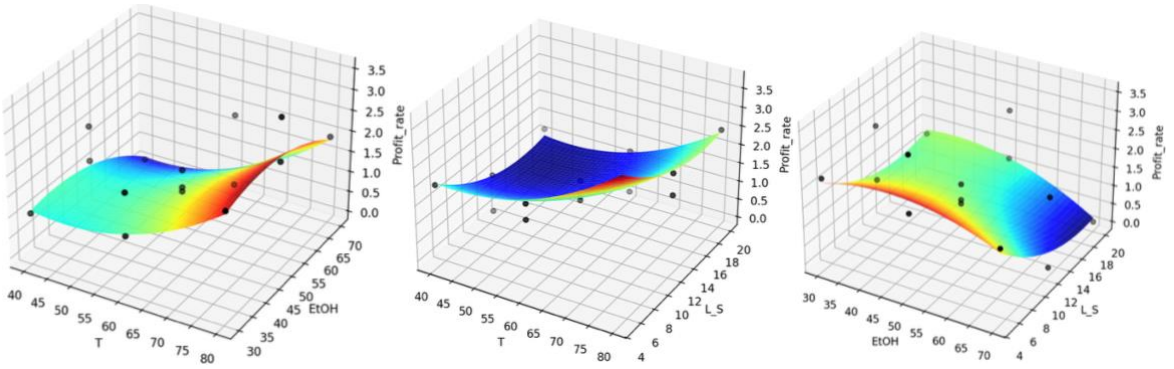

Figure 9. Response surfaces of Box-Behnken design (T = temperature [°C], LS = L/S ratio [ml/g], EtOH = ethanol concentration [vol-%]). Blue and red areas denote the lowest and highest values, respectively.

Table 7. Box-Behnken model parameters.

Parameter	Value
R^2	0.976
Adjusted R^2	0.932
F value	22.3
p value	0.0016
Q^2	0.781

Table 8. Box-Behnken model coefficients.

Coefficient	Value	p value
Intercept	1.28	<0.05
T	0.56	<0.05
EtOH	-0.29	<0.05
LS	-0.43	<0.05
T^2	0.28	<0.05
$EtOH^2$	-0.23	<0.05
LS^2	0.25	<0.05
T*EtOH	0.06	0.42
T*LS	-0.03	0.71
EtOH*LS	-0.1	0.19

The program also calculated the optimal factor values using a numerical optimizer. The optimal values for the highest profitability were T = 80 °C, EtOH = 47.5 vol-%, and L/S = 5 ml/g. Nevertheless, it is important to consider that optimal values for the highest polyphenol yield were markedly different (EtOH = 55 % and L/S = 20). Higher L/S ratio and ethanol concentration enable slightly higher yields, but they also significantly increase production costs (ethanol price). On the contrary, while higher temperature increases energy demand, the higher polyphenol yield offset the increased production cost. Therefore, its linear coefficient is positive while the linear coefficients for L/S ratio and EtOH% are negative (Table 8).

The factors affect the profit rate in the following order: temperature > L/S ratio > EtOH%. However, each factor has a significant effect on the profit rate. It is also important to note that each factor has a significant quadratic effect, but none have dependencies on each other; all interaction terms are insignificant ($p > 0.05$). This means that changing one factor would not significantly affect the other factors' optimal values. Overall, the model showed a good fit to the experimental data, with an adjusted R^2 of 0.932. The high Q^2 value of 0.781 further indicated a good predictive ability. The model is statistically significant ($F = 22.3$, $p = 0.0016$), confirming that the Box-Behnken model adequately explain the response within the investigated experimental region (Table 7).

6.3.2 Full Factorial RSM

The response surface of Full Factorial design is shown in Figure 10. The response is profit rate (section 5.6). Model parameters are shown in Table 9 and model coefficients in Table 10.

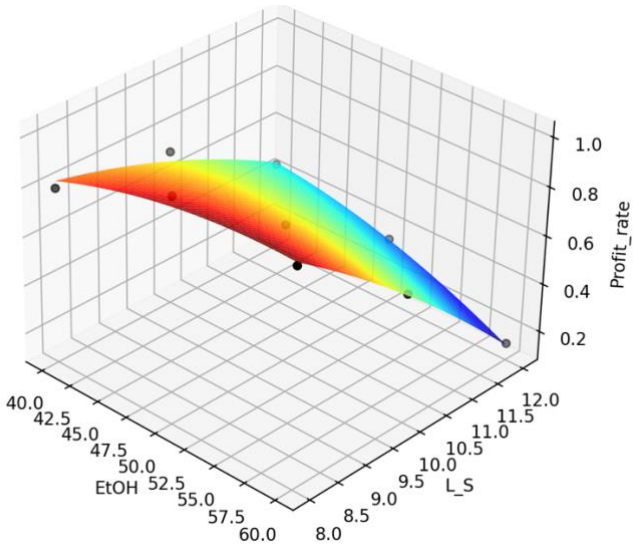

Figure 10. Response surface from Full Factorial design (EtOH = ethanol concentration [vol-%], LS = L/S ratio [ml/g]). Blue and red areas denote the lowest and highest values, respectively.

Table 9. Full Factorial model parameters.

Parameter	Value
R^2	0.990
Adjusted R^2	0.973
F value	58.2
p value	0.0035
Q^2	0.909

Table 10. Full Factorial model coefficients.

Coefficient	Value	p value
Intercept	0.69	<0.05
EtOH	-0.03	0.14
LS	-0.25	<0.05
$EtOH^2$	-0.03	0.21
LS^2	-0.03	0.29
EtOH*LS	-0.08	<0.05

As in the Box-Behnken optimization, the program calculated the optimal factor values for the Full Factorial model using a numerical optimizer. The optimal values were EtOH = 57.4 vol-% and L/S = 8 ml/g (the minimum value). Again, pure polyphenol yield was shown to almost linearly increase with ethanol concentration and L/S ratio. Nevertheless, the yield increase was insufficient to compensate the increasing ethanol consumption which increases the production costs and therefore decreases profit rate. Therefore, the linear coefficient for L/S ratio is negative (Table 10).

Although the L/S ratio was the only significant factor, the model showed a great fit to the experimental data and great predictive ability (adjusted $R^2 = 0.973$; $Q^2 = 0.909$). The model is statistically significant with F = 58,2 and p = 0.0035 (Table 9). Nevertheless, the high indicator values may be caused by the presence of only two significant coefficients in the model. The lower significance may be attributed to the narrower experimental region compared to the Box-Behnken design. This results in smaller changes in the response within the experimental region and therefore poor signal-to-noise ratio.

Interestingly, in the Full Factorial model EtOH*LS coefficient was significant: changing the liquid-to-solid ratio may change the optimal ethanol concentration. However, the coefficient value is minor (-0.08) and therefore could be considered noise. It can be a consequence of random errors in the extraction process and analyses. Alternatively, the change in L/S ratio may affect the concentration gradient and therefore solubility of the compounds. Another possible explanation is that while the dry peel preferentially absorbs water rather than ethanol, the effective ethanol concentration in the recovered extract may change as a function of the L/S ratio. This could affect polyphenol solubility in the extract. Nevertheless, since the yield differences were small, and the average trend seems to favor higher ethanol concentration, it can be concluded that optimal ethanol concentration is most likely within 50 and 60 %.

6.3.3 Bioactive profile analysis

Bioactive profile (HRE, dry), obtained from HPLC, is shown in Figure 11.

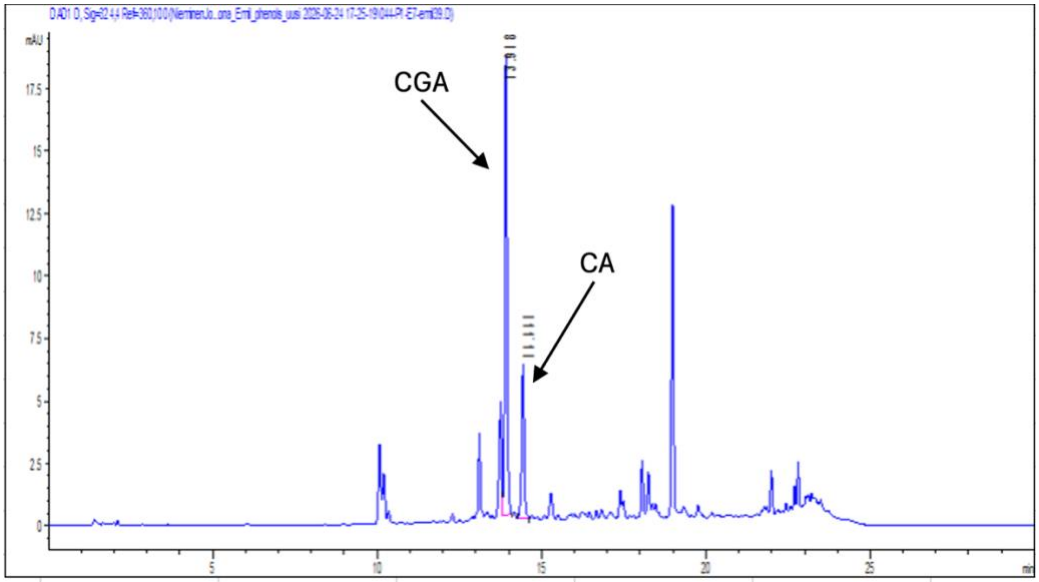

Figure 11. Chromatogram of centrifuged potato peel extract obtained from dried peel.

The detected compounds are chlorogenic acid and caffeic acid (14- and 14.5-min retention, respectively). The high peak at 19 min is likely ferulic acid or p-coumaric acid (Wu et al., 2012). α-chaconine is not shown in the Figure 11 because it was assayed in a separate run. Gallic acid standard was also run, and it eluted at around 3.9 min. A minuscule peak can be observed in the chromatogram at that location, suggesting the gallic acid concentration is insignificant. The minor concentration may also reveal why the Folin-Ciocalteu method, where gallic acid was used as a standard, was inaccurate compared to the HPLC results. Nevertheless, while different compounds absorb the DAD wavelength (280 nm) at different intensities, the compounds other than CGA, CA, and α-chaconine, which were used as HPLC standards, cannot be quantified. Moreover, different compounds may elute at the same time, resulting in overlapped peaks in the chromatogram. While diode array (DAD) detector cannot separate or identify compounds, mass spectrometer (MS) detector is recommended for more reliable assay.

HPLC results are shown in Table 11. All concentrations are in [mgGAE/gdb] (TPC) or [mg/gdb] (HPLC results). TPC denotes total phenolic content (Folin-Ciocalteu result), and CGA is Chlorogenic acid, CA is Caffeic acid, and Cha is α-chaconine (HPLC results).

Table 11. HPLC results in [mgGAE/gdb] or [mg/gdb].

Sample	TPC	CGA	CA	Cha
BB4	5.02	0.14	0.12	1.19
BB5	5.16	0.19	0.16	0.96
BB6	5.19	0.18	0.19	1.49
BB8	6.46	0.25	0.23	1.71
BB13	4.21	0.20	0.16	1.43
BB14	4.13	0.18	0.14	1.17
Dry, fine	3.79	1.01	0.39	1.65
Dry, fine, wash	0.98	0.17	0.07	0.16
Wet, coarse	2.40	0.97	0.20	0.59
65 °C	4.82	0.73	0.16	0.76
80 °C	5.56	0.75	0.16	1.03
1st extract	2.20	0.26	0.12	0.76
3rd extract	0.07	0.01	0.00	0.04
1st extract	2.28	0.28	0.13	0.82
2nd extract	0.32	0.04	0.01	0.11
60% EtOH	2.04	0.24	0.11	0.66
HRE control	2.43	0.16	0.11	N/A
UAE control	2.76	0.17	0.13	N/A

The HPLC assay revealed specific remarks. First, dry peel seems to extract relatively more glycoalkaloids than phenolic acids, although the concentration of both increased compared to wet. The same trend of relatively higher glycoalkaloid extraction can be observed with increased temperature and ethanol concentration. Second, the relative glycoalkaloid-to-CGA ratio is prominent. Potato peel should contain more phenolic acids compared to glycoalkaloids (Table 2). Besides, phenolic acids are more polar and should dissolve better into hydroethanolic solvent than glycoalkaloids. The reason remains unknown. Possible explanations are sprouting in the potato peel sample which increases glycoalkaloid concentration, or isomerization or degradation of CGA into products the HPLC did not detect.

Singh et al. (2011) noted that increasing temperature increases total polyphenol yield but also changes the composition. The HPLC results confirm increased glycoalkaloid co-extraction with temperature. While higher temperature decreases solvent polarity, and glycoalkaloids are less polar than polyphenols, the increased solubility is reasonable.

6.3.4 Effect of temperature

Temperature was the most significant factor affecting the yield and profit rate in the Box-Behnken (BB) model ($p < 0.05$). Higher temperature increases polyphenol solubility, and reduces solvent surface tension and viscosity, which increase solvent penetration (Wang et al., 2020). It also promotes the cell wall disruption. Therefore, higher temperature is expected to increase the yield, until thermal degradation occur. Since the extractions were conducted at under 80 °C and only for 30 minutes, the thermal degradation could not be observed (Figure 9). Nevertheless, while higher temperature increases chlorogenic acid (CGA) yield, the increase is relatively lower compared to the TPC increase. This suggests more significant glycoalkaloid and sugar co-extraction at higher temperatures. Higher temperature may also increase Maillard reaction products, inflating TPC. Therefore, the purification process economics and desired purity level may limit the decision on optimal temperature over TPC or chlorogenic acid yield.

6.3.5 Effect of liquid-to-solid ratio

Liquid-to-solid (L/S) ratio was the second most affecting factor ($p < 0.05$). Increasing L/S ratio increases concentration gradient; the actual driving force in extraction (Singh et al., 2011). When considering pure yield, higher L/S ratio increased the response in both Box-Behnken (BB) and Full Factorial (FF) models; optimal L/S value was the highest in both. However, considering the increase in ethanol cost and larger volume to be processed when more solvent is used, the increased yield was counterbalanced and the effect turned around: in both BB and FF models, the optimal profit rate was achieved at the lowest L/S ratios. The L/S ratio coefficient was significantly negative in both models (Tables 8 and 10), indicating the added ethanol and processing volume at higher L/S ratio strongly affects the process economics.

6.3.6 Effect of ethanol concentration

Ethanol concentration had a negative parabolic effect in both models (Figures 9 and 10). Therefore, optimal concentration can be found from the vertex. While the optimal

concentration was notably different according to BB model (47.5 vol-%) and FF model (57.4 vol-%), the models had different ethanol concentration ranges (30-70 vol-% for BB, 40-60 vol-% for FF). In addition, the ethanol concentration was not significant in the FF model (both EtOH and $EtOH^2 > 0.05$). Therefore, the profit rate was not significantly influenced by ethanol concentration within the narrower experimental region of 50 % ± 10 %. Narrow range also increases the effect of random error.

When polyphenol yield was used as a response instead of profit rate, higher ethanol concentration was beneficial. Ethanol has higher diffusivity than water, denoting faster extraction kinetics. However, the additional ethanol cost seems to compensate this effect, and therefore the profit rate remains approximately constant within the 50 % ± 10 % range. At broader range (50 % ± 20 %, Box-Behnken model), the parabolic effect is clearer. This indicates that hydroethanolic mixture containing approximately half ethanol and half water has the best solvation power for polyphenols. The parabolic effect is also supported by the literature (Table 3), where ethanol concentration of 55 – 60 vol-% has typically shown optimal yields.

Based on HPLC analysis, higher ethanol concentration slightly increased glycoalkaloid co-extraction. As in the case of higher temperature, higher ethanol concentration also reduces the solvent polarity. The observation is therefore plausible, considering the lower polarity of glycoalkaloids compared to phenolic acids.

6.3.7 Effect of extraction time

Initially, extraction is expected to occur rapidly, due to the dissolution of readily accessible bioactive compounds from the cell surface. As the extraction proceeds, the release of intracellular compounds becomes increasingly limited by diffusion, resulting in a slower increase in yield. After an optimal extraction time, the yield is expected to reach equilibrium. Under prolonged extraction or severe processing conditions, the yield may decrease because of thermal and oxidative degradation of the bioactive compounds, as well as their possible back-adsorption onto the solid matrix.

The hypothesis is well shown in the results. In the kinetic experiments (Figures 6, 7, 8) the yield was already approximately 4 mgGAE/gdb at the first point (10 min), and it gradually

increased with time. Further, Figure 8 shows lower yield at 90 minutes compared to 40 min. The experiments were conducted at 75 °C, and thermal degradation is probable when the time was extended to 90 min. Moreover, the liquid may have become saturated in polyphenols during the first 30 minutes, preventing further yield increase with time. The experiment employed a low L/S ratio of 10 ml/g, and a significant portion of the volume was absorbed into the peel.

Faster mass transfer kinetics at high temperature (Fig. 6) and in dry process (Fig. 7) are also plausible. Higher temperature increases water and ethanol diffusivity (according to Arrhenius equation). Drying pretreatment, in turn, is expected to increase the biomass porosity, rupture the cell matrix, and physically shorten the diffusion path. Further, while literature (Table 3) shows pure water to be inferior to hydroethanolic solvent, the water inside the wet peel might have impaired the diffusion compared to dried peel where the hydroethanolic solvent migrates into the cell structure. Likewise, according to the Fick's second law, higher concentration gradient increases mass transfer speed. The concentration gradient is expected to be greater in dried peel because the fresh solvent does not initially contain dissolved polyphenols. In wet peel, intracellular water already contains dissolved polyphenols, reducing the driving force for mass transfer.

Figure 8 confirms the expected extraction behavior, with the UAE yield increasing over time. Therefore, the increase followed by a decrease in UAE yield observed in Figure 5 is more likely attributable to noise, sampling error, or loss of polyphenols during sample settling than actual desorption and back-adsorption of the polyphenols during the extraction. Figure 8 also shows that the initial extraction kinetics were faster for UAE than for HRE, although the HRE achieved a comparable yield within 30 minutes.

6.3.8 Liquid retention after extraction

Four extraction sets were conducted to determine the location of unextracted polyphenols (Figure 12). In the first three experiments the extraction time was 10 minutes, and the last experiment (rightmost in the Figure 12) was 30 min to isolate the effect of extraction time. The experiments show the portion of extracted polyphenols (the first extraction of each experiment) compared to the unextracted polyphenols that were either trapped in the cell matrix (leftmost experiment in the Figure 12) or that were trapped in the retained liquid (the

two middle experiments in the Figure 12). Further, the rightmost extraction (Figure 12) shows the effect of extraction time on the recovered polyphenols. The retained liquid denotes the extraction solvent that could not be recovered by solid/liquid separation (Buchner filtration) but remained in the solid phase due to the dry peel absorbing the solvent. The symbols 1st, 2nd, and 3rd denote first, second, and third extractions, while the wash, wash1, and wash2 denote water washing. In each extraction, 10 ml of solvent (50 % ethanol) was used, and in each wash 10 ml of water was used. The response is TPC [mgGAE/gdb].

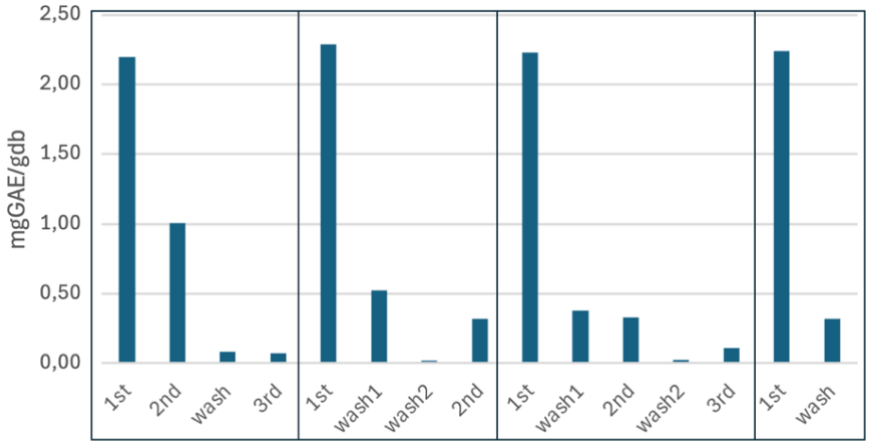

Figure 12. Extraction trial to determine polyphenols trapped in the retained liquid.

The experimental set reveals that most polyphenols were extracted during the 10-min extraction, and of the unrecovered portion, significant amount was in the retained liquid. The set also shows that one washing step is sufficient, while two consecutive extraction steps combined with a washing step recovers 96 % of the polyphenols. HPLC results revealed similar trend, confirming that the TPC was sufficient to estimate the CGA concentration in this experiment. Further, solvent recovery using centrifugation was conducted as a reference, and it did not recover more solvent compared to Buchner filtration. However, the experiments did not include mechanical filter press which may reduce the amount of trapped liquid. Moreover, while up to half of the polyphenols are present in esterified or bound forms in the peel matrix (Rodríguez-Martínez et al., 2021), moderate enzymatic pretreatment prior to extraction is expected to further increase the yield. While enzymes may depolymerize the desired substances, the treatment should be controlled and the enzymes precisely selected.

6.3.9 Optimized extraction conditions

Both temperature and ethanol concentration increased chlorogenic acid yield but also glycoalkaloid co-extraction. To balance the chlorogenic acid to glycoalkaloid (CGA-to-glycoalkaloid) ratio, the optimal values obtained from the BB and FF models change slightly: approximately 60 °C and 50 % ethanol can be deemed optimal, in contrast to 80 °C and 55 % based on TPC alone. With an L/S ratio of 8 ml/g, the production cost (section 5.6) was estimated at 11.88 €/$kg_{extract}$, with a throughput of 0.289 $kg_{extract}$/h/$kg_{dry_raw_material}$. The extract is assumed to contain 10 wt-% of polyphenols. The production cost is nearly two times lower than that of the unoptimized process (Figure 4), and the CGA-to-glycoalkaloid ratio is also more favorable. Although the TEA does not include fixed overhead costs, the process appears economically viable, assuming a selling price of polyphenol extract to be 20 €/$kg_{extract}$ (Jin et al., 2021). Moreover, Jin et al. (2021) concluded that a biorefinery plant producing both polyphenols and biochar was more economically viable compared to a plant only producing polyphenols from grape pomace. Similarly, because the extracted potato peel residue is rich in carbohydrates, its conversion into bio-oil and biochar through pyrolysis could further increase the process economics while simultaneously reducing the waste stream. While many potato processors already utilize the peels in the production of fertilizer or biogas (Martínez-García et al., 2024), the added extraction and pyrolysis steps may generate significant additional income without major fixed capital investment.

Further, the chlorogenic acid yield difference between dry and wet extraction was smaller compared to what TPC suggested. While the finding is noteworthy, the inconsistent batches and the difficulty to reduce wet peel particle size without starch discharge, which compromised the subsequent filtration trials, make wet processing impractical.

7 Discussion

This chapter discusses the process practices and economics. The upstream and downstream processes, extraction methods, and analytical procedures are examined sequentially.

7.1 Process practices

To achieve technical and economic feasibility, the entire process flow must be considered. This subchapter discusses the upstream and downstream process remarks which transpired during this work.

The phenolic yield is affected not only by the drying, extraction, and recovery processes but also by the peeling and peel handling. Potatoes are typically washed thoroughly before and during peeling, while the water-soluble phenolic compounds may leach into the wash water. Consequently, a significant fraction is already lost in the peeling stage. Potatoes are peeled with two different techniques, both of which consume water during the process. Steam peeling produces a thinner, creamy texture, and it is more concentrated in the phenolic compounds. In contrast, abrasive peeling removes a thicker layer which contains more underlying parenchyma tissue, resulting in lower phenolic concentration. However, the higher temperature during steam peeling may cause thermal degradation of the phenolic compounds. Moreover, if not processed promptly after peeling, the phenolic acid content in the peel may decrease, whereas the glycoalkaloid content may increase (Almeida et al., 2023).

Regarding the pretreatment method, drying followed by sufficient particle size reduction enabled the highest yield and fastest filtration. The effect of drying was isolated by comparing dry and wet processes at similar particle size, while the effect of particle size was evaluated by comparing fine and coarse milling of the dry peel. Both factors contributed to the polyphenol yield: drying increased the yield even at comparable particle size, and finer milling further enhanced the yield. In addition to achieving higher yield, drying the peel before extraction enables easier solid-liquid separation, more consistent results, easier particle size reduction, less volume to be processed in the purification stages, and lower energy consumption during product recovery and drying. These results were consistent with

previous literature (Almeida et al., 2024). When selecting the drying equipment, it is important to note that industrial drum heaters might create hot spots, whereas a belt heater is easier to control and may be more energy-efficient compared to drums (Green, 2025).

However, significantly more glycoalkaloids were co-extracted from the dry peel, and small peel particles may blind the filter cloth. Therefore, glycoalkaloid removal and narrow particle size distribution are important for ensuring continuous downstream processing and consistent batches while keeping glycoalkaloid levels within permissible limit. To prevent fine dust and consequent blinding of the filter press cloth during solid-liquid filtration, a knife mill is recommended. It enables a narrower particle size distribution and produces less heat compared to a hammer mill, also decreasing the risk of thermal degradation during the milling step.

Further, low liquid-to-solid ratio and sufficient ethanol recovery are crucial for economics. Distillation followed by membrane filtration is one option to recover the solvent, while distillation alone significantly impairs the process economics according to the TEA. Similarly, the solid-liquid separation after extraction may employ centrifugation, distillation, or crystallization instead of filtration. In this case the wet process, which could not be filtrated, also becomes technically feasible. However, polyphenol back-adsorption may occur during centrifugation, and it should be carried out at the extraction temperature or immediately after heat exchange to recover the extraction heat. Cake washing is also more complex to integrate into a decanter centrifuge, and that device increases considerably both investment and maintenance costs of the process compared to a filter press. Therefore, the process economics calculation requires pilot-scale experiments with accurate energy consumption measurements, and estimations for investment and maintenance costs throughout the process flow. Such metrics can then be fed into the TEA model to determine the actual viability of the wet process compared to dry process.

To further increase the extraction yield, polyphenols present in esterified and insoluble bound forms within the peel matrix must first be released. These forms account for approximately half of the total polyphenol content (Rodríguez-Martínez et al., 2021). Their release requires disruption of the cell matrix through fine milling, enzymatic pretreatment, and long extraction times. Although enzymatic pretreatment is increasingly studied and several patents have been filed (Espacenet, 2026), its industrial application remains limited due to high cost of enzymes, their limited stability, and long treatment times required.

7.2 Method comparison

This work did not find ultrasound-assisted extraction (UAE) to be superior to heat reflux extraction (HRE) at any extraction temperature. Although the UAE conditions were not further optimized, it remains unclear whether a potentially higher yield would justify its higher operation costs and process complexity. While UAE achieved 14 % higher total yield compared to HRE (Table 11), the HPLC revealed CGA concentration to improve by only 5 %. This finding is compatible with literature (Kumari et al., 2017). Moreover, UAE produced less consistent results and appeared to co-extract more cell debris than HRE. While ultrasonic cavitation physically disrupts cells and therefore enables faster kinetics, it also co-extracts fine particles, colloids and aggregates to which polyphenols remain attached. These are difficult to recover and may be lost during solid-liquid separation.

Some studies did not identify temperature as a significant factor in UAE (Riciputi et al., 2018), while others reported a moderate effect (Wang et al., 2020). In this work, both extraction methods significantly benefited from higher temperature. Higher temperature enables lower solvent viscosity, higher polyphenol solubility, and more effective cell disruption. These mechanisms seem to promote extraction kinetics also in UAE, although its primary extraction mechanism is ultrasonic cavitation rather than heating. Consequently, UAE requires both ultrasonic and thermal energy, exceeding the energy demand of HRE, especially at scale. Furthermore, the experimental results (Figure 12) suggest that a 2-stage HRE can achieve extraction kinetics comparable to those of UAE. For both methods, a 10-min residence time was sufficient.

7.3 Analytical constraints

HPLC results (Table 11) showed high glycoalkaloid concentrations: in most samples, the assayed α-chaconine concentration was 2-10 times higher compared to that of chlorogenic acid (CGA). Although Table 2 shows that glycoalkaloid concentrations vary more than CGA concentration and can exceed those of CGA, the literature (Table 2) generally concludes CGA to be predominant. However, the CGA-to-glycoalkaloid ratio strongly depends on factors such as potato variety, light exposure, and storage conditions and duration. Moreover, peeling stimulates glycoalkaloid biosynthesis (Torabian et al., 2023). Since the peels were

not extracted and analyzed immediately after peeling, glycoalkaloid concentrations may have increased during storage.

CGA and α-chaconine concentrations ranged from 0.2-1 and 1-1.7 mg/gdb, respectively. The values are within the ranges reported in literature: previous studies (Table 3) have assayed CGA concentrations of 0.1-4 mg/gdb, and synthesis studies conclude CGA and α-chaconine concentrations of 0.17-6 and 0.07-27 mg/gdb, respectively (Table 2). Likewise, the caffeic acid (CA) concentration (0.1-0.4 mg/gdb) was within the reported range of 0.16-3 mg/gdb (Table 2). Nevertheless, a purification step is essential for the extraction process, because the glycoalkaloid concentrations exceeded the approved limits of 0.2 mg/gdb total glycoalkaloids (Khanal et al., 2024). Without purification, the number of applications for the extract decreases.

The results show that TPC (total phenolic content, Folin-Ciocalteu method) was not a reliable indicator of CGA and CA concentrations (Table 11). For example, increasing extraction temperature from 65 to 80 °C increased TPC by over 15 %, while CGA concentration remained unchanged. The increase in TPC is likely caused by increased co-extraction of glycoalkaloids and sugar, and formation of Maillard reaction products. Similarly, dry extraction produced a 1.58-fold higher TPC than wet extraction, but only 1.04-fold higher CGA concentration.

ABTS and DPPH assays may be used instead of the Folin-Ciocalteu (FC) assay. Unlike FC, they measure radical scavenging (antioxidant capacity) rather than reducing capacity. They are less susceptible to interference from ascorbic acid and reducing sugars and may therefore reflect the polyphenol concentration more accurately. Both assays are based on a reaction between polyphenols and a stable free radical, resulting in a measurable color change. However, each assay induces a different reaction mechanism with phenolic compounds. Instead of direct quantification, they measure specific antioxidative attributes of the mixture of compounds present in the sample (Rumpf et al., 2023). Likewise, gallic acid was used as the calibration standard in the FC assay. While its concentration was low in the samples, using CGA as the calibration standard instead might provide more reliable and consistent results. Nevertheless, HPLC remains critical to quantify the individual compounds, while coupling HPLC with mass spectrometer (MS) provides more reliable identification than diode array detector (DAD).

According to Table 11, no evidence of thermal degradation of CGA was observed when the extraction temperature was increased from 65 to 80 °C. The experiment was conducted in duplicate. While the CGA concentration remained almost unchanged, the CA concentration did not increase either. Under harsh conditions, CGA can hydrolyze into its constituents, CA and quinic acid, which is expected to increase the concentration of CA in the sample. Instead, increasing the ethanol concentration appears to extract relatively more CA, and less CGA. This observation is consistent with the increased extraction of glycoalkaloids at higher ethanol concentrations, considering that CA is less polar than CGA.

The design of experiments showed that temperature, L/S ratio, and ethanol concentration all significantly affect the yield and process profitability. Considering that HPLC revealed the increased temperature and ethanol concentration to favor the extraction of glycoalkaloids relative to CGA, these parameters should therefore be selected based not only on extraction yield, but also on the desired composition of the extract. Because temperature had a minor effect on CGA concentration, lower extraction temperature and ethanol concentration should optimize CGA recovery while minimizing glycoalkaloid co-extraction. The HPLC results showed poor CGA recovery at 40 °C, indicating that the optimal extraction temperature is likely between 40 and 65 °C. Lower temperature also decreases the process energy demand, increasing profitability. However, because less polar compounds dissolve better at higher temperatures and ethanol concentrations, the process parameters must be optimized separately for each compound of interest.

8 Conclusions

In this Thesis, a process for extracting polyphenols from industrial potato peel waste was developed and optimized. A practical approach and techno-economic feasibility were prioritized throughout the work. The objectives were to screen two extraction methods, ultrasound-assisted extraction (UAE) and heat reflux extraction (HRE), together with two pretreatment options, drying and milling or milling only, and to optimize temperature, ethanol concentration, and liquid-to-solid (L/S) ratio for a favorable CGA-to-glycoalkaloid ratio and profitability. Two experimental designs were employed, with profit rate as the response.

The most economical and technically feasible combination was HRE with the dry process. The key factors affecting the CGA-to-glycoalkaloid ratio were successfully identified and optimized: process economics was especially sensitive to L/S ratio and temperature, whereas drying, and high temperature and ethanol concentration increased glycoalkaloid co-extraction. The optimal profit rate was achieved with 55-60 % hydroethanolic solvent, 80 °C (upper limit), and an L/S ratio of 8 ml/g (lower limit). However, to maximize chlorogenic acid (CGA) yield while simultaneously minimizing glycoalkaloid co-extraction, 50 % ethanol and 60 °C are recommended. A short extraction time of 10 minutes was sufficient, especially with a two-stage extraction process including a washing step. Such a process recovered 96 % of the extractable polyphenols.

Based on the results, the following process is proposed as the most economical and technically feasible way to recover polyphenols from industrial potato peel waste: belt drying, knife milling, and two-stage CSTR extraction. The extraction includes a washing step and immediate solid-liquid separation to recover the product, and it is operated at 60 °C, 10 min per stage, 50 % hydroethanolic solvent, and an L/S ratio of 8 ml/g. This process achieves a total phenolic content (reducing capacity) of 4.82 mgGAE/gdb, including 0.73, 0.16, and 0.76 mg/gdb of CGA, CA, and α-chaconine, respectively. The results are within the ranges reported in the literature.

The production cost of the optimized process was estimated at 12 €/$kg_{extract}$. Nevertheless, the cost excludes fixed overhead costs, and large-scale operation would be necessary to improve economic viability. The economics may improve further if a biorefinery approach,

including extraction and subsequent pyrolysis of the solid residue, is applied. The process also generates wastewater rich in minerals which can be valorized as fertilizer. Such a biorefinery would produce polyphenols, biochar, bio-oil, and fertilizer, while contributing to a more circular, zero waste process and simultaneously greater profitability for the potato processors.

The results agree with previous studies: extraction is generally improved by higher temperature, smaller particle size and, to some extent, by ethanol concentration. However, this Thesis added three contributions: it identified the significant role of L/S ratio in process economics, the significant role of temperature and ethanol concentration on the extract composition, and it showed that while UAE achieves a slightly higher yield, it is not necessarily superior to HRE due to its greater complexity, co-extraction of fine particles, and higher energy demand. Future UAE research should therefore optimize for economic performance and selectivity rather than yield alone, and conduct efficient reactor design and pilot-scale experiments. Moreover, the recovery of polyphenols from the co-extracted fine particles must be addressed.

More broadly, the results suggest that industrial potato peel waste is a promising raw material for polyphenol production due to its abundance, low cost, and high content of chlorogenic acid; a well-studied polyphenol with significant anti-diabetic, anti-obesity, antioxidant, and cardioprotective effects. CGA has already attracted practical use in several areas, including nutraceuticals, functional foods, cosmetics, pharmaceuticals, and animal feed, which still rely mostly on synthetic antioxidants and require inexpensive and sustainable sources of CGA. Moreover, valorizing industrial potato peel waste contributes to circular economy and reduction of food waste. This Thesis contributed to the process development by identifying and optimizing the core variables of a technically and economically feasible process. It also showed that, while novel extraction methods such as UAE remain attractive, their low technology readiness level currently limits their industrial use. Therefore, their optimization against techno-economic indicators, rather than pure polyphenol yield alone, is important for increasing their practical readiness.

9 Limitations and recommendations for future research

The work was limited to a laboratory-scale batch process. Therefore, the technical and economic viability of the process involves a significant degree of uncertainty. Pilot-scale production using the optimized parameters, including purification of the polyphenol extract and pyrolysis of the solid residue, is required to reduce this uncertainty and obtain more accurate techno-economic indicators.

Besides the pilot validation, future studies must address three additional process-related aspects. First, glycoalkaloid separation and extract purification remained uninvestigated. An adsorption step to purify CGA could be included and simulated to assess its economic viability. While glycoalkaloids are often deemed undesirable owing to their cytotoxicity, a cation ion exchange resin placed before polyphenol purification could instead recover them as a separate value stream, as glycoalkaloids are positively charged at neutral and low pH. Second, controlled enzymatic pretreatment before drying may increase polyphenol yield by breaking ester bonds linking polyphenols to the cell matrix. Otherwise, a significant fraction remains bound in the residue. Third, energy integration is crucial to design and implement throughout the process to offset the high drying and extraction temperatures.

In addition to techno-economic analysis, life-cycle assessment (LCA) is also necessary to identify not only the most profitable but also environmentally friendly process. A more sustainable process might also improve market potential, since the product could appeal to a broader range of customers. Moreover, the Folin-Ciocalteu assay was shown to correlate weakly with CGA concentration; ABTS or DPPH assays, or calibration against CGA instead of gallic acid, are recommended for future studies. HPLC, preferably coupled with a mass spectrometer detector, is recommended for reliable quantification of individual phenolic acids and glycoalkaloids. Finally, the short residence time of microwave-assisted extraction (MAE), with a frequency lower than the commonly used 2.45 GHz, may achieve efficient polyphenol extraction; albeit the ATEX-rated equipment and frequent magnetron replacement currently limit its technical and economic feasibility.

Overall, this Thesis contributed to the development of an industrially viable route for valorizing potato peel waste. The results provided a basis for further work toward a practical and economically viable process to produce polyphenols.

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