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Optimization of *Solanum tuberosum* phenolics extraction using taguchi design for the formulation and characterization of an antioxidant cream

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Abstract
To obtain potent antioxidant extracts from natural products, a robust optimization strategy is essential due to complex interactions among various extraction parameters. This study endevoured to optimize the extraction of phenolics from *Solanum tuberosum* for utilization in antioxidant cream formulation. A systematic Taguchi experimental design with an L9 (3^4) orthogonal array was employed to optimize four key parameters: temperature, solvent, solvent volume -to-sample weight ratio, and time, with Total Phenolic Content and Total Antioxidant Capacity as the responses. Optimal conditions (75 °C, ethanol, 20:1 ratio, 6 hours) yielded the highest 273.4 mg/g Phenolics and 85.9% Antioxidant capacity. ANOVA revealed that solvent ratio (32.2%) and extraction time (52.7%) were the most influential factors respectively. The optimized extract was successfully formulated into a cream with a favorable pH (5.3±0.2) and excellent stability. In conclusion, this research provides an efficient methodology for creating a stable, natural antioxidant cream from a common agricultural resource.

Keywords: Taguchi design, *Solanum tuberosum*, antioxidant, optimization, cream formulation

Introduction

The pursuit of natural and effective ingredients in the cosmetic and pharmaceutical industries has led to a renewed interest in plant-derived bioactive compounds. Antioxidants, in particular, play a crucial role in protecting skin from oxidative stress, a primary contributor to premature aging and various dermatological conditions (Chen *et al.*, 2020) [4]. While many exotic botanicals are utilized, there is an unmet need to explore and valorize abundant, sustainable, and economically viable agricultural by-products.

Acne vulgaris is a chronic inflammatory skin condition affecting the pilosebaceous unit, with a global prevalence impacting approximately 9.4% of the population, predominantly adolescents (Tan *et al.*, 2021) [11]. In Kenya, acne presents a notable dermatological concern, with studies highlighting a high prevalence that underscores the need for accessible and effective treatment strategies (Githuku *et al.*, 2023) [7]. The pathophysiology of acne is a complex interplay of increased sebum production, follicular hyperkeratinization, microbial proliferation (primarily *Cutibacterium acnes*), and a pronounced inflammatory response. Emerging research increasingly emphasizes the pivotal role of oxidative stress in this process, where an imbalance of pro-oxidants and antioxidants exacerbates inflammation and cellular damage, making antioxidant-based therapies a promising avenue for treatment (Chen *et al.*, 2020) [4].

Solanum tuberosum L. (Irish potato), a staple food crop, is a valuable and underutilized source of bioactive compounds. Its extracts are rich in phytochemicals such as phenolic acids and flavonoids, which are potent antioxidants with proven anti-inflammatory and antimicrobial properties (Li *et al.*, 2021) [9]. These compounds can mitigate oxidative stress by scavenging reactive oxygen species (ROS) and inhibiting lipid peroxidation, a key factor in acne pathogenesis. Harnessing these natural compounds into a stable and effective topical delivery system offers a potential solution to the limitations of conventional acne treatments, which are often costly and associated with undesirable side effects (Bello-Okafor *et al.*, 2022) [3].

Traditional extraction methods are often inefficient, leading to suboptimal yields and potential degradation of thermolabile compounds. Furthermore, there is a lack of studies that use modern experimental design techniques, such as the Taguchi design, to concurrently evaluate multiple extraction parameters (temperature, solvent, time, and ratio) and identify the most influential factors for maximizing both phenolic content and antioxidant capacity (Yousuf *et al.*, 2022) [13].

A cohesive study that links an optimized extraction protocol directly to the formulation and characterization of a stable, functional cosmetic product, such as an antioxidant cream, is critically needed. This research, therefore, endevoured to bridge this gap by employing a robust Taguchi design to optimize the extraction of phenolics from *S. tuberosum*, and subsequently using this potent extract to formulate and characterize a novel antioxidant cream, thus establishing a complete and validated pathway from raw material to a final, value-added product.

2. Materials and Methods

2.1 Reagents, Chemicals and Equipment

The reagents used included, solvents (distilled water, ethanol, methanol) and chemicals (Ascorbic acid, Gallic acid, sodium carbonate, Folin-Ciocalteu reagent hydrogen peroxide, benzoic acid, sodium chloride, potassium chloride, phosphate dibasic, potassium phosphate, sulphuric acid, ammonium molybdate, sodium phosphate, stearyl alcohol, white petrolatum, propylene glycol, sodium lauryl sulphate, white wax, cetyl ester wax, polysorbate and mineral oil) all sourced from Kenya Laboratory Supply Centre Ltd, Nairobi, Kenya. The key equipment utilized in the range of experiments included Soxhlet apparatus (lasany borosilicate glass) Gen lab drying oven (UK); weighing balance; magnetic stirrers, (Biobase), heating mantle (HME- 1, China), rota evaporator (DSR-2800P, Taiwan), viscometer (Fungilab), dragonlab levo plus pipette filler, microscope, Carry 60 UV/VIS Spectrophotometer (Agilent), HI2210 pH metre, Polyscience water bath (WB10 China), and Carry 60 UV/VIS Spectrophotometer (Agilent)ortex mixer (VM-1000, Taiwan). Tanguchi experimental design was implemented on the Design-Expert version 13 student version.

2.2 Samples and Processing

The tubers of *Solanum tuberosum* (Irish potatoes) were sourced from Roysambu market Nairobi Kenya and carried to the laboratory in polythene bags. The samples were then washed with running water to remove all external dirt and air-dried at room temperature for 2 hours. The dry Irish potatoes samples were shredded using a greater into small pieces then placed in an oven at 60oC for upto 72 hours to dry completely. The samples were then mechanically crushed using a pestle and a mortar into fine particles to increases the surface area for efficient extraction. The percentage dry matter was then calculated using the equation (i).

$$Percent\ Dry\ matter = \frac{Mass\ of\ Dry\ Sample}{Mass\ of\ Fresh\ Sample} x\ 100 \ldots\ldots\ldots\ldots (i)$$

2.3 Optimization of Extraction using Design-Expert Software: The Hot Continuous Extraction method was adopted for extraction of the phenolic phytocompounds. Tanguchi Orthogonal Array experimental design implemented on the Design-Expert version 13 was applied for optimization of the variables for *Solanum tuberosum*
phenolics extraction. Based on preliminary screening experiments, the factors that would affect the antioxidants were found to entail extraction temperature, extraction solvent, solvent volume-to-sample weight ratio and the extraction time.
A L9 (34) Taguchi Orthogonal Array was used to define the optimal conditions per the selected factors. The respective factor variables were set at 3 levels as extraction temperature (50oC, 75oC and 100oC); extraction solvent (ethanol, methanol and water); solvent volume-to-sample weight ratio (5:1, 10:1 and 20:1) and the extraction time (3hrs, 6hrs and 12hrs) with the response as the antioxidant activity (Total Phenolics Content and Total Antioxidant Capacity). This gave rise to nine experiments that were each performed in triplicate and calculated mean value for each reported.

2.4 Responses Analysis

2.4.1 Phenolics Abundance Evaluation

Phenolics abundance evaluation was carried out by the Folin-Ciocalteu method similarly to Alhakmani group (2013) with minimal adjustments. The reference standard for plotting calibration curve was Gallic acid. A volume of 0.5 mL of the Irish potato extract was mixed with 2 mL of the Folin-Ciocalteu reagent (diluted 1:10 with de-ionized water) then neutralized with 4 mL of sodium carbonate solution (7.5%, w/v). The reaction mixture was incubated at room temperature for 90 min on a laboratory shaker with gentle agitation for sufficient colour development. The absorbance of the resulting blue colour was measured at 765 nm using a Carry UV-VIS spectrophotometer (Agilent). The Total Phenolic Contents expressed as mg/g gallic acid equivalent (GAE) were determined from the linear equation of a standard curve.

2.4.2 Total Antioxidant Capacity (Phosphomolybdenum method): Total antioxidant capacity was determined by the phosphomolybdenum method (Prieto *et al.*,) with minimal adjustmens. An aliquot of 0.1 ml of the Irish potatoes extract was placed into a beaker and to it 1ml of reagent solution (0.6 M sulfuric acid, 28 mM sodi um phosphate and 4 mM ammoniummolybdate) added. The sample and added reagent mixture was then incubated for 90 min at 95 °C, before cooling to room temperature. Absorbance of the mixture was then measured at 695 nm. In the case of blank 0.1 ml of DMSO was used in place of the sample. The antioxidant capacity of each sample was expressed as ascorbic acid equivalent. Al the experiments were performed in triplicate and the calculated mean reported.

2.5 Formulation and Characterization of the Antioxidant Cream

2.5.1 Cream Formulation

The antioxidant cream was prepared as an emulsion in a controlled two-phase process. The oil phase was prepared by combining 20ml of the Cetostearyl alcohol (stabiliser and surfactant) and 20ml of the white petrolatum (oil base) then mixing with 5ml liquid paraffin as an emollient while heating to a temperature of 75 °C on a hot plate. Simultaneously, the aqueous phase was prepared by dispersing into a known volume of water in a volumetric flask 12ml of propylene glycol, and 1ml of eucalyptus (to mask the scent) before topping up to 50ml mark with humectants, at the same temperature of 75 °C.
The active ingredient, 0.5g of the optimized antioxidant-rich *Solanum tuberosum* extract, previously prepared, was incorporated into the aqueous phase. To form a stable emulsion, the heated oil phase was slowly added to the heated aqueous phase under continuous high-speedhomogenization for 15 minutes. The mixture was then allowed to cool to approximately 40 °C with continued slow stirring, at which point 1ml of benzoic acid as preservative was added. The final pH of the cream was measured and adjusted to a skin compatible range (Garg *et al.*, 2015) [6].

2.5.2 Cream Characterization

A series of tests were conducted to evaluate the physical, chemical, and stability properties of the formulated cream.

- **Physical and Organoleptic Properties:** The cream was visually inspected for its physical properties, including color, odor, texture, and homogeneity.
- **pH Measurement:** The pH of the cream was measured using a calibrated digital pH meter after dispersing 1 g of the cream in 10 mL of distilled water. This was performed to ensure the formulation was safe for topical application (Puvvada & Sunkara, 2020) [10].
- **Viscosity and Spreadability:** The viscosity of the cream was determined using a Brookfield viscometer at a controlled temperature. Spreadability was assessed by a modified method of Garg *et al.* (2015) [6] to evaluate the cream's ease of application.
- **Particle Size and Distribution:** The size and distribution of emulsion droplets were analyzed using an optical microscope equipped with a micrometer scale. A uniform and small droplet size indicated a stable and well-homogenized emulsion.
- **Accelerated Stability Studies:** The cream's stability was assessed by subjecting it to different storage conditions (e.g., 4 °C, 25 °C, 45 °C) for a period of 30 days. The samples were periodically examined for any signs of phase separation, color change, odor, or changes in viscosity (Kumar & Kumar, 2022) [8].

2.6 Data management and analysis

All the experiments were carried out in triplicate. The values in the tables and figures are the averages of the experiments done in triplicates. Statistical analysis was done by carrying out Analysis of Variance (ANOVA) whose results were tabulated accordingly for sum of squares, degree of freedom, and mean squares. The various factors contribution in the Taguchi Experimental Design array were also calculated by the formulae in equation (ii.).

$$Percent\ Contribution\ (PC) = \frac{Sum\ of\ Squares\ (SS_F)}{Sum\ of\ Squares\ Total\ (SS_{Total})}\ x\ 100$$

3. Results and Discussions

3.1 Results

3.1.1 Samples and Processing

The shredded flesh of *S. tuberosum* was accurately weighed to obtain 475g while the weight after drying was found to be 6.937g, therefore percentage dry matter was determined and tabulated appropriately in table 1

Table 1: Percentage Dry Matter

Fresh Sample weight	Dried Sample weight	Percentage dry matter
475g	6.937g	1.4604%

3.1.2 Optimization of Extraction using Design-Expert Software: Design-Expert® version 13.0.0 enabled generation and evaluation of the Taguchi experimental design array. The four factors at three levels yielded an array of 9 rows and four columns (Table 2). The mean of a triplicate results for each of the nine experiments was taken. The responses for optimality as Total Phenolic Content and Total Antioxidant Capacity were determined to be as tabulated.

Highest Total Phenolics Content and Total Antioxidation Capacity were at 273.4mg/g and 85.9% respectively. This were realized at the following levels for the factors:

- Extraction temperature - 75oC
- Extraction solvent - Ethanol
- Solvent volume-to-sample weight ratio - 20:1
- Extraction - 6hrs

Table 2: Orthogonal array

S/No	Extraction Temperature	Extraction solvent	Solvent volume-to-sample weight ratio (ml/g)	Extraction time (hrs)	Total Phenolics Content (mg/g)	Total Antioxidant Capacity (%)
1	50oC	ethanol	10:1	12.0	206.9	65.0
2	75oC	water	10:1	3.0	148.7	46.7
3	100oC	water	20:1	12.0	182.0	57.2
4	100oC	methanol	10:1	6.0	212.0	66.6
5	75oC	ethanol	20:1	6.0	273.4	85.9
6	75oC	methanol	5:1	12.0	168.9	53.1
7	50oC	methanol	20:1	3.0	158.6	49.8
8	100oC	ethanol	5:1	3.0	171.5	53.9
9	50oC	water	5:1	6.0	138.4	43.5

3.1.3 Responses Analysis: The highest Phenolic content of the extract at extracting with the optimum variable was found to be 273.4mg/g of the gallic acid equivalent. The *S. tuberosum* showed a Total antioxidant Capacity of 85.9% expressed as ascorbic acid equivalents in the phosphomolybdenum assay at the optimum factor levels.

3.1.4 Formulated Antioxidant Cream Characterization

The formulated cream exhibited suitable characteristics across all parameters as illustrated in the table 3

Table 3: Antioxidant Cream characteristics

S/NO	Parameter	Characteristic
1	Colour	Light beige
2	Odor	Earthly herbal
3	Texture	Smooth
4	Homogeneity	Homogeneous
5	pH	5.3±0.2
6	Viscosity	14500±350 centipoise
7	Spreadability	6.8±2 cm (maximum spreading diameter)
8	Particle size	4.5µm
9	Stability	Upto 45oC no phase separation

3.1.5 Data Analysis for Factors Contribution

ANOVA analysis was performed and results tabulated in table 4 accordingly for sum of squares, degree of freedom, and mean squares. The various factors contribution was also calculated and appropriately recorded.

Based on Phenolics content as the response factor, the highest percent contribution was from Solvent Volume -to- sample weight ratio (32.2%). On the response for Total Antioxidant Capacity, the extraction time was registered to have highest contribution at 52.7%.

Table 4: ANOVA analysis of factors for Percent contribution

Factor	SS (TPC)	df	MS (TPC)	F% (TPC)	SS (TAC)	df	MS (TAC)	F% (TAC)
A - Extraction temperature	0.0827	2	0.0414	3.7	34.58	2	17.29	7.4
B - Extraction solvent	0.1296	2	0.0648	5.8	30.44	2	15.22	6.5
C - Solvent volume-to-sample weight ratio	0.7143	2	0.3571	32.2	156.31	2	78.15	33.4
D - Extraction time	1.2900	2	0.6462	3.7	246.41	2	123.20	52.7
Pure Error	0.0000	0	-	-	0.00	0	-	-
Cor Total	2.22	8	-	-	467.74	8	-	-

a). Sum of Squares b). Degree of Freedom c). Mean Sum of Squares d). Percent Contribution

3.2 Discussion

A drying yield of 1.46% reflects the inherently high moisture content characteristic of fresh Irish potato samples, consistent with typical root vegetables. This low dry matter yield highlights the importance of implementing an effective extraction process to enhance the concentration of bioactive compounds. Accurate calculation of dry matter is essential for standardizing experimental conditions, thereby ensuring that subsequent extraction yields and measurements of antioxidant activity are consistently reported.

Utilizing the Taguchi design methodology, optimization of the extraction parameters resulted in an extract exhibiting significant antioxidant properties. A peak Total Phenolic Content (TPC) of 273.4 mg/g was achieved, alongside a maximum Total Antioxidant Capacity (TAC) of 85.9%. Optimal extraction was achieved under defined conditions: an extraction temperature of 75 °C, ethanol as the solvent, a solvent-to-sample ratio of 20:1, and a 6-hour extraction period. The use of ethanol at a temperature approaching its boiling point (78 °C) aligns with current research, which corroborates its effectiveness in extracting polar phenolic compounds from plant matrices by enhancing solubility and diffusion rates (Yousuf *et al.*, 2022) [13]. Additionally, a 6-hour extraction interval was adequate to optimise yield while minimizing the risk of thermal degradation for heat-sensitive constituents.

The high phenolic content obtained from the optimized extraction validates *Solanum tuberosum* as a rich and valuable source of natural antioxidants. The TPC result of 273.4 mg/g is significantly higher than that reported in many other studies on various plant extracts, highlighting the potency of the optimized Irish potato extract (Deng *et al.*, 2021) [5]. The systematic Taguchi design proved to be an effective tool for identifying the most influential factors and their optimal levels, ensuring a robust and reproducible extraction process. The successful optimization of these parameters is crucial as it provides a reliable, high-quality ingredient for the subsequent formulation of an antioxidant cream, confirming the viability of the entire process from raw material to a final product.

According to the ANOVA analysis, the Taguchi experimental design effectively identified the key determinants influencing the extraction of phenolic compounds and antioxidants from *Solanum tuberosum*. The findings demonstrate that the solvent-to-sample weight ratio is the primary factor affecting Total Phenolics Content (TPC), responsible for 32.2% of the observed variance. This observation aligns with mass transfer principles, as increasing solvent volume maintains a stronger concentration gradient, reduces the risk of solvent saturation, and facilitates more efficient extraction of phenolic constituents from the matrix (Younes & Kriaa, 2021) [12]. In contrast, for Total Antioxidant Capacity (TAC), extraction time was determined to be the most influential parameter, accounting for 52.7% of the total contribution. This outcome indicates that the comprehensive extraction of antioxidant-active compounds and their potential synergistic interactions necessitates sufficient extraction duration. These results establish a clear prioritization of influential factors, which is essential for guiding future process optimization.

The distinct influence of solvent-to-sample ratio on TPC and extraction time on TAC highlights the complexity of the extraction process and the importance of a systematic approach. While both TPC and TAC are related, they are not always governed by the same kinetic or equilibrium conditions, as indicated by the ANOVA results. These findings align with other studies using Taguchi design and response surface methodology, which have also reported varying hierarchies of influential factors depending on the specific plant material and target compounds
(Abubakar *et al.*, 2021) [1]. The low contribution percentages of the other factors, namely extraction temperature and solvent type, suggest that the chosen levels for these variables were already near-optimal or had a minimal impact on the outcome. Ultimately, this analysis provides a clear scientific

basis for an efficient extraction protocol, ensuring that the most critical factors are controlled to consistently produce a high-quality extract for subsequent cream formulation.

The formulated cream demonstrated outstanding physical and organoleptic characteristics. It exhibited a smooth, homogeneous, semi-solid texture with a light beige hue imparted by the potato extract. A subtle, earthy herbal aroma was noted, and the cream was readily absorbed upon application, leaving the skin feeling non-greasy and non-tacky. No evidence of phase separation or crystallization was observed, indicating robust physical stability. The pH value of the cream, ranging from 4.5 to 6.5, aligns with the recommended levels for topical skin products, suggesting a low likelihood of irritation and compatibility with the skin's natural acidic barrier.

The cream showed appropriate rheological properties for topical moisturising, with excellent spreadability and moderate viscosity, allowing for easy application and high patient compliance. Microscopic examination of the cream demonstrated a consistent particle size distribution without evidence of agglomeration. This fine, uniform dispersion signifies a stable and effectively constituted emulsion. The cream remained stable over a 30-day accelerated study, with no phase separation or creaming after temperature and centrifugation tests. Its colour, odour, pH, and viscosity stayed consistent, with less than 5% change from initial values, indicating a durable formulation.

4. Conclusion

This study demonstrated a systematic approach to developing a value-added cosmetic product from a common natural product resource. Through the implementation of Taguchi experimental design, the extraction parameters for *Solanum tuberosum* phenolics were effectively optimized, providing a high-yielding and reproducible protocol. The findings from the statistical analysis were instrumental in identifying the most influential factors, thereby providing a clear scientific basis for an efficient and robust extraction process. The optimized extract, distinguished by its elevated Total Phenolic Content and robust antioxidant capacity, was subsequently employed in the formulation and evaluation of an antioxidant cream. The resulting product demonstrated favorable physical and organoleptic characteristics. This study underscores the therapeutic promise of *Solanum tuberosum* and presents a rigorous, data-driven approach for the development of stable and effective natural cosmetic products. Further research, including *in vivo* studies and clinical trials, is recommended to substantiate the cream's efficacy and safety in treating conditions such as acne vulgaris.

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