

Innovative Model Two-Phase Makeup Remover Containing of Jerusalem Artichoke Tubers Extract

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Abstract

In response to the growing demand for high-quality cosmetics, it is observed an increasing interest in products containing plant extracts and we are obliged to look for products will be a rich reservoir of many active substances. The study analyzed the effect of the concentration of Jerusalem artichoke tubers (*Helianthus tuberosus L.*) extract on properties of model two-phase makeup remover.

The extract was obtained with the use of ultrasound. The antioxidant properties of plant material were evaluated using the DPPH method and the content of phenolic compounds and flavonoids in the extracts was determined. Cytotoxicity of the examined extracts on fibroblast cells was also examined. The next, formulation and technologies for the production of products differing in the content of plant extract were developed. 4 specimens were analyzed with a different content of extracts: 0%, 5%, 15% and 15%. As a result of the conducted analyzes, it was shown that the extracts are characterized by strong antioxidative properties and positively affects the proliferation of fibroblast cells.

The inserting of this extract into a cosmetic product did not change their stability. As a result of the analyzes carried out, it was found that the model make-up removing cosmetics are safe to use - after performing epidermal patch tests, none of the analyzed preparations caused changes on the skin. The obtained results indicate that the analyzed Jerusalem artichoke tubers extracts are a valuable sources of active ingredients exhibiting antioxidant activity, not negatively affecting the functional properties of the model two-phase makeup remover.

Keywords: antioxidants, cytotoxicity, Jerusalem Artichoke, two-phase makeup remover

INTRODUCTION

Nowadays, when practically everything can be synthetic, people are looking for natural ways to effectively protect and care for the skin, keeping it fresh and young for as long as possible. For this reason, the cosmetics industry is increasingly focused on acquiring and using natural cosmetics of the highest quality for the production of cosmetic preparations. Considering the wide range of plant materials with high biological activity, phenolic compounds are most valued as secondary products of plant metabolism. They are present in the leaves, flowers, roots and stems of higher plants [1]. Phenolic compounds include phenolic acids, flavonoids and tannins. Due to the complex chemical structure, phenolic acids and flavonoids are characterized by a wide range of biological activity, and thus have antioxidant properties. This property is characterized by the ability to remove reactive oxygen species (ROS). Anti-inflammatory, antifungal and antibacterial effects have also been proven [2]. The task of phenolic compounds with antioxidant properties is to prevent the harmful effect of excessive amounts of free radicals arising from oxidative stress on skin cellular structures. Phenolic compounds, such as polyphenols, are readily incorporated into the redox reaction, thereby inhibiting the oxidation of fatty acids that are components of cosmetics. Currently, plants with antioxidant properties constitute the largest group of ingredients for skin care products [3].

Jerusalem artichoke (*Helianthus tuberosus L.*) is a plant native to North America. It belongs to the family of complex Compositae (*Asteraceae*) and is related to

sunflower [4]. He was brought to Europe in 1612 as edible, and decorative plant. Initially, it was widespread in France and Germany. The discovery of Jerusalem artichoke is due to Samuel Champlain - a French traveler. He noticed that the Brazilian Indian tribe "Topinambu" eats potato-like plants. He found that tubers had a sweetish taste reminiscent of an artichoke. He moved them to Canada, and then they came to Europe as a delicacy [5]. In turn, in ancient China, the anti-inflammatory, analgesic and antipyretic effects of this plant were discovered, so it was used to treat wounds, swelling and fractures [6]. Jerusalem artichoke tubers are the most desirable part of the plant and are harvested at the end of autumn, because during this period sugars are moving from the upper parts to the underground [7]. Jerusalem artichoke tubers consist of about 80% water, 3% protein and 15-16% carbohydrates. The largest part of carbohydrates, over 80% is inulin, as well as starch and simple sugars. In addition, the tubers of this plant contain vitamin B1, iron, potassium, magnesium and copper. They are also a valuable source of exogenous amino acids such as threonine and tryptophan [8] In addition, the presence of phenolic compounds in tubers has been proven and is on average 221.0 mg 100 g⁻¹ [9]. Phenolic acids in Jerusalem artichoke tubers constitute 16.6% of dry matter [10]. Active polyphenol compounds, known as effective radical scavengers, protect against UV radiation, strengthen the connective tissue of the skin and the walls of blood vessels [11]. That is why care products based on *Helianthus tuberosus L.* tubers are becoming more and more popular and have found application in the production of cosmetics.

MATERIALS AND METHODS

1.1. Plant material and extraction procedure

Helianthus tuberosus L. were planted in a sunny position in the March. The tubers were planted in a depth of 10–15cm. The distance between rows was about 90 cm. The tubers of Jerusalem artichoke were collected from the region of Subcarpathian Voivodeship in Poland during the August 2018. The collected tubers were transported to the laboratory and prepared for analysis.

The fresh plant extracts were prepared by using ultrasound-assisted extraction method (UAE) performed according to the method described by Ying et al. (2011) [12]. In ultrasonic bath (Digital Ultrasonic Cleaner) equipped with time controller. About 30 g of Jerusalem artichoke tubers were packed to the glass tubes and extracted with a 300ml of water in room temperature. The mixture was homogenized for 48 min (8 cycles for 6 min). Then, obtained extracts were collected and filtered through Whatman filter paper No. 10. Tuber extract was stored in the dark in 4°C for subsequent analysis.

1.2. Total phenolic content determination

The total phenolic content of tubers *Helianthus tuberosus L.* extract was determined spectrophotometrically by the Folin-Ciocalteu method according to the procedure reported by Singleton et al. with some modifications [13]. The 300µL of leaves extract solutions and 1500µL of 1:10 Folin-Ciocalteu reagent were mixed and after 6min in the dark, 1200µL of sodium carbonate (7.5%) was added and to each samples. After incubation in the dark at the room temperature absorbance at 740nm was measured (Helios Aquamate by Thermo Scientific). The total phenolic concentration was calculated from a gallic acid (GA) calibration curve (10–100mg·mL⁻¹). Data were expressed as gallic acid equivalents (GA)·g⁻¹ of extract averaged from three independent measurements.

1.3. Total flavonoids content determination

The total flavonoid content of analyzed extract was evaluated using aluminium nitrate noanhydrate according to the procedure reported by Matejić *et al.* with modifications [14]. Briefly, 600µL of plant extracts solutions was mixed 2850 µL of reaction mixture (80% C₂H₅OH, 10% Al(NO₃)₃ × 9H₂O and 1M C₂H₃KO₂). After 40min of incubation in the dark at 25°C, the absorbance at 415 nm was measured spectrophotometrically by Thermo Scientific spectrophotometer. The total flavonoids concentration in extracts were calculated from a quercetin hydrate (Qu) calibration curve (10–100mg·mL⁻¹) and expressed as mg of quercetin equivalents (QU) in 1L of extract averaged from three independent measurement.

1.4. DPPH radical scavenging assay

The ability to scavenge free radicals by Jerusalem artichoke extracts obtained from tubers was evaluated using DPPH (2,2-diphenyl-1-picrylhydrazyl) assay according to the method described by Brand-Williams *et al.* [15]. 167µL of 4mM ethanol solution of DPPH was mixed with 33µL analyzed samples in different concentrations (0,25% - 10%). The absorbance of the remaining DPPH radical was measured at λ=517 nm in every 5 minutes for 30 minutes on UV-Vis

spectrophotometer Filter Max 5 (Thermo Scientific). DPPH solution mixed with equal volume of distilled water was served as a control. The percentage of the DPPH radical scavenging were calculated using the equation (Eq. 1):

$$\%DPPH \bullet \text{scavenging} = \frac{[Abs \text{ control} - Abs \text{ sample}]}{Abs \text{ control} \times 100\%} \quad (1)$$

1.5. Cell Culture

BJ cells (fibroblasts, ATCC@CRL-2522™) used in the experiments was obtained from the American Type Culture Collection (Manassas, VA 20108, USA). Fibroblast were maintained in a MEM (Minimum Essential Medim, Gibco) contains Earle's salt and L-glutamine, supplemented with 5% (vol/vol) FBS (fetal bovine serum, Gibco), and 1% (vol/vol) antibiotic (100 U·mL⁻¹ Penicillin and 1000µg·mL⁻¹ Streptomycin, Gibco). All cultured cells were kept at 37°C in a humidified atmosphere of 95% air and 5% of carbon dioxide (CO₂). When the cells reached confluence, the culture medium was removed from the flask (VWR) and cells were rinsed two times with sterile PBS (Phosphate-Buffered Saline, Gibco). The confluent layer was trypsinized using Trypsin/EDTA (Gibco) and then resuspended in fresh medium. Cells were treated with varying concentrations (0,25, 0,5, 1, 2,5, 5, 10%) of aqueous tuber and *Helianthus tuberosus L.* extract.

Cell viability assay

Alamar Blue Assay

The resazurin sodium salt (AlamarBlue) (Sigma, R7017), was used to assess fibroblast cell viability. Cells were seeded in transparent 96- well plates at a density of 1×10⁴ cells/well with fresh MEM medium and exposed to different concentration (0,25 – 10%) of tested *Helianthus tuberosus L.* extracts for 24h. The control group were unexposed cells maintained in a MEM medium. After exposure, resazurin solution was transferred into the plates for a final volume 250 µL/well and final concentration of 60µM resazurin. Then, the cells were incubated for 2 hours at 37°C in darkness. The absorbance was measured at the wavelength λ=570 nm using a microplate reader (FilterMax F5, Molecular Devices). Measurements were repeated three times for each Jerusalem artichoke extracts concentration. Results were expressed as a percentage of the viability of the control sample versus the control (100%).

Technology for Obtaining Prototypical two-phase makeup remover

Based on prior experiences, prototypical two-phase makeup remover containing the previously prepared aqueous *Helianthus tuberosus L.* extract was developed. The final concentrations of the extract in the model formulations were 5, 10 and 15%. The formulations meet the requirements applicable to natural cosmetics. The composition of prototypical natural two-phase makeup remover formulations are listed in Table 1

Table 1. Formulation of model two-phase makeup remover.

Ingredient (INCI)	Ingredient Content			
	Base [wt %]	Sample 1 [wt %]	Sample 2 [wt %]	Sample 3 [wt %]
Aqua	45,4	40,4	35,4	30,4
Capric/Caprylic Triglycerides	50,0	50,0	50,0	50,0
<i>Helianthus tuberosus L.</i> extract	0	5,0	10,0	15,0
Sodium Chloride	1,0	1,0	1,0	1,0
Sodium benzoate	0,6	0,6	0,6	0,6
Panthenol	1,0	1,0	1,0	1,0
Glycerin	2,0	2,0	2,0	2,0

In our research four samples of prototypical two-phase makeup remover were obtained. The preparation method was the same for all the model samples. At the beginning, demineralized water Sodium Benzoate, Glycerol and Panthenol were poured into a beaker. The Capric/Caprylic Triglycerides were weighed in a second beaker. The content of both beakers were mixed. Then the previously prepared aqueous *Helianthus tuberosus L.* extracts and Sodium Chloride were added. The all ingredients were mixed until the completed dissolved (mechanical stirrer Chemland O20, 200 rpm, 15 min).

1.6. Epidermal patch tests

The study was conducted among 25 healthy women, aged between 19 and 27 years. The mean age of the participants was 23 years. None of the participants had taken any medications for at least one week prior to undergoing epidermal patch tests. The tests were performed by a person qualified in conducting epidermal patch tests.

The first stage of the patch tests was the so-called open test with two-phase makeup remover (model formulations). The formulation was applied to the face of the study's participants in designated locations. The first reading of the tests was performed after 15 minutes, and then after 30, 45 and 60 minutes, followed by readings performed 2, 6 and 24 hours after the application. If no allergic reaction occurred in a given subject within a period of 24 hours, epidermal patch tests were conducted [16, 17, 18].

The epidermal patch tests were carried out using IQ Ultra test chambers from Chemotechnique Diagnostics. A patch test unit consists of two rows of five chambers (10 test chambers in total) mounted on adhesive carrier tape. The tests were performed with eight chambers in which previously prepared model formulations were placed (a set of gels diluted in water at a ratio of 1:2 and a set of undiluted gels). In accordance with the manufacturer's recommendations, 30 µl portions of hapten were also added to each chamber [16, 18].

The epidermal patch tests were made in accordance with guidelines of the International Contact Dermatitis Research Group. Two identical hapten-containing patches were applied to the skin of the back (between the scapulas) of the test participants. The first patch was removed after 24 hours, while the second one (in case of no allergic reaction) – after a further 24 hours. The findings were interpreted in accordance with the recognized international system for recording epidermal patch test results [16].

The scale for the interpretation of patch test results:

- (negative) – no reaction

+ (weakly positive) – erythema and oedema

++ (positive) – erythema, oedema, papules or vesicles

+++ (strongly positive) – erythema, oedema, papules or vesicles spreading beyond the area of hapten adherence

IR – irritant reaction, including erythema without oedema, large vesicles, extravasations, pustules [16,17]

1.7. pH measurements

The pH levels of the cleaning formulations under study were determined by direct measurements in the test samples using a pH meter (Elmetron CPS 411). During the measurement samples were mixed using a magnetic stirrer.

1.8. Statistical Analysis

Each value is the mean of three replicates. Values of different parameters were expressed as the mean ± standard deviation (SD). The one-way analysis of variance (ANNOVA) and Bonferroni posttest between groups were performed at the level *P* value of <0.05 to evaluate the significance of differences between values. Statistical analyses were performed using GraphPad Prism 5.0 (GraphPad Software, Inc., Sand Diego CA).

RESULTS AND DISCUSSION

Phenols and flavonoids compounds of natural origin represent a broad group of secondary plant metabolites. These substances have received attention because of their physiological function, mainly effected by antioxidant, antimicrobial and anti-inflammatory properties [19-22]. It has been recognized that flavonoids show antioxidant activity and their effects on human nutrition and health are considerable. The mechanisms of action of flavonoids are through chelating or scavenging process [23, 24]. Phenolic compounds are a class of antioxidant agents which act for free radical terminators [25]. Plants polyphenols are formed by two basic cycles of metabolism, in the acetic acid cycle and shikimic acid cycle. In the plant material, these substances may occur in both a simple form and a compound with a high degree of complexity. As a result, the composition of the phenolic fraction is strongly depended on the variety, agrotechnical and climatic conditions, but most of all on the part of the plant from which the raw material was obtained. The synergistic activity of many ingredients contained in the Jerusalem artichoke tubers makes it a valuable component of cosmetic products. The amount of compounds obtained

during the extraction process depends mainly on the time and temperature of extraction, the plant material to solvent ratio, and the extraction type [26, 27]. Ultrasound-assisted extraction (UAE) seems to be a right method, capable to extract high quantities of bioactive compounds in a short time. Therefore, in this work we used UAE method to separate flavonoids and phenolic compounds from tubers of *Helianthus tuberosus L.* [28].

To determine the biological activity of *Helianthus tuberosus L.* tubers extract we examined total phenolic content (TPC) and total flavonoid content (TFC). The results for Jerusalem artichoke extract obtained from ultrasound-assisted extraction (UAE) are presented in Table 2.

Table 2. Total phenolic and total flavonoids content in aqueous extract (UAE -ultrasound-assisted extraction) of *Helianthus tuberosus L.* tubers. Values are mean of six replicate determinations (n=6) ± SD. The concentrations of the tested extracts are 0,25, 0,5, 1, 2,5, 5, 10%.

Total phenolic content [mg GA/g dry weight ± S.D.]	Total flavonoids content [mg Qu/g dry weight ± S.D.]
30,57 ± 2,67	4,96 ± 0,56

The result showed, that tubers extract exhibits the high phenolic and flavonoid content, compared to the different extracts of plants. It also was observed, that the TPC and TFC content were increased with increasing extract concentration. The result also showed that the total content of phenols and flavonoids depends on the type of solvent used for the extraction [29].

It has been shown that obtained extract is a rich source of compounds which have a biologically activeness, thus we hypothesised that *Helianthus tuberosus L.* tubers also might exhibit antioxidant properties. DPPH is an easy, fast and sensitive method to investigate the antioxidant activity of a particular compound or plant extracts [30]. In this case we have been check the antioxidant potential of obtained extract. The six different concentrations (0,25; 0,5; 1; 2,5, 5 and 10%) were evaluated by DPPH• scavenging assay, where reaction is based on changing colour of free radical extract following to incubation with analyzed substances. The decrease of absorption values after adding tested substance to the radical solution is directly proportional to the number of formed DPPH•. Based on obtained results it has been noticed that Jerusalem artichoke tubers aqueous extract shows antioxidant activity. The potency to scavenge free radicals was directly dependent on used concentration [31].

Considering the concentrations of used extracts, a different dependence of the DPPH inhibitory capacity was demonstrated. The highest scavenging free radical activity was observed for 10% concentration of examined *H. tuberosus L.* extract. In this case the decrease of free radical amount rich to 70% compare to the control. This concentration was several times more effective than lower concentrations. It was also observed that the ability to inhibit DPPH • was directly related to the extract concentration. The DPPH inhibitory potency increases

with increasing extract concentration. The dependence of scavenging free radicals from time has also been observed. It has been shown that after a certain period of time the percentage of scavenging of free radicals increases. Considering the different concentrations, it can be noticed that after 30 minutes at lower concentrations (0.25, 0.5, 1, 2.5%) the percentage of scavenging of free radicals is small, while at concentrations of 5 and 10% after 30 minutes the ability to inhibit DPPH increased to 60 and 70%.

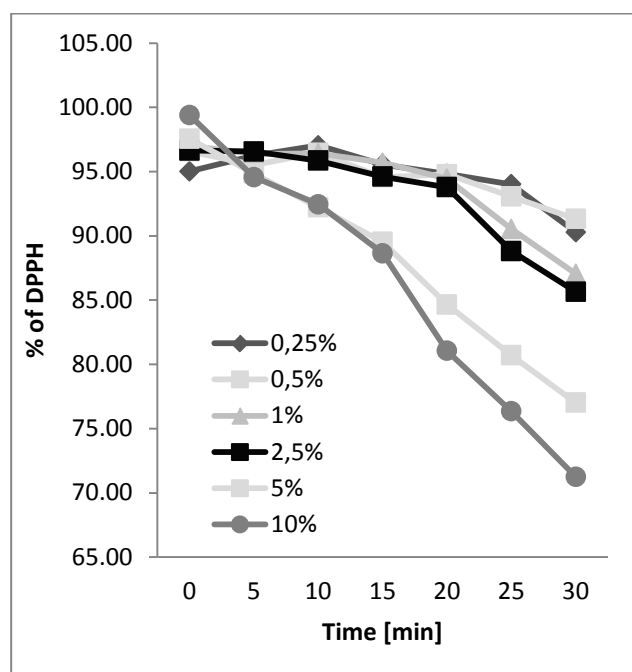


Figure 1. Kinetics of the absorbance changes in DPPH• solutions in the presence of various concentrations of aquaous extract of *Helianthus tuberosus L.* tubers. Values are mean of three replicate determinations (n = 3).

Alamar Blue dye is redox indicator commonly used to evaluate cellular health and metabolic function. The Alamar Blue bioassay has been used by many researchers to assess cell viability and cytotoxicity of different types of compounds in vitro. Alamar blue test is based on fluorometric detection of metabolic mitochondrial activity of the examined cells. This test measures changes in the fluorescence of the dye in the intracellular environment and detect the number of metabolic active cells [25, 32].

Our results obtained using the Alamar Blue assay showed that Jerusalem artichoke extract in all tested concentration (0,25 - 10%) did not show cytotoxic effect on fibroblast cells. In addition, the induction of proliferative effect of UAE extract was observed. The highest increase of cell proliferation, resulting from the largest number of metabolic active cells, was noticed for 2,5% of tested extracts (Figure 2). In case of that resazurin reduction is the result of multiple metabolic reactions and it is not a direct indicator of the mitochondrial dysfunction, we think that this bioassay is an appropriate tool for assessing cellular health and viability. Considering the observed

tendency, indicating that with increasing concentration of the extract above 2,5% cell proliferation is decreasing, it may be assumed that concentrations above 10% may have cytotoxic effects on fibroblast cells.

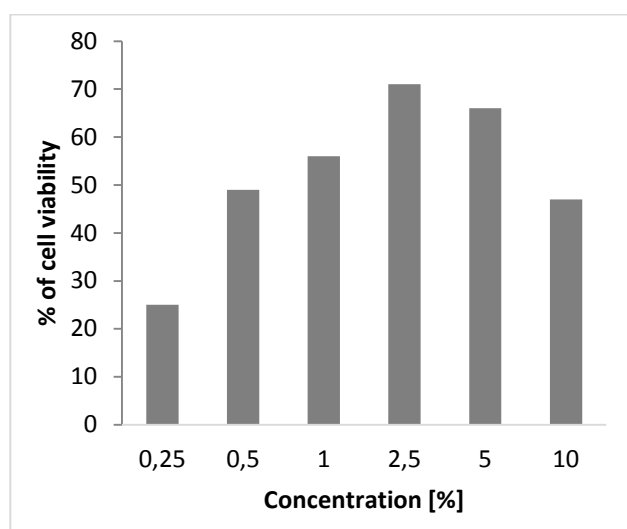


Figure 2. The effect of increasing concentrations of aqueous UAE *Helianthus tuberosus* L. extracts (0,25, 0,5, 1, 2,5, 5, 10%) on Alamar blue dye uptake in cultured fibroblasts after 24h of exposure. Data are the mean \pm SD of four independent experiments, each of which consists of three replicates per treatment group. *** $p < 0.001$ versus the control (100%).

The pH of the four model preparations was measured. The results are presented in the table 3.

Table 3. Ph measurements of two-phase makeup remover.

Base	Sample 1	Sample 2	Sample 3
6,0	5,7	5,4	5,1

It was observed that the pH of two-phase makeup remover was directly related to the extract concentration. As the increase of Jerusalem artichoke extract concentration in the preparation, the pH decreased. However, the pH of the makeup remover is similar to the pH of the skin.

Epidermal patch tests

As the open test demonstrated no skin reaction, epidermal patch tests were performed. There were no signs of skin irritation in any of the testers after the removal of the first patch. The same observation were obtained after the removal of the second patch.

CONCLUSION

The result of the present study showed that the extract of *Helianthus tuberosus* L., which contain high amount of flavonoid and phenolic compounds, exhibited the greatest antioxidant activity. The high scavenging property of Jerusalem artichoke may be due to hydroxyl groups existing in the phenolic compounds chemical structure that can provide the necessary component as a radical scavenger. The high antioxidant capacity of *Helianthus*

tuberosus L. extracts may indicates potential use as valuable ingredient in the cosmetic industry.

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