

## **A food-grade antioxidant production using industrial potato peel by-products**

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**Abstract.** Currently, industrial potato processing waste recycling and re-use is an important topic in the food industry, but no actual processing facilities could be found at the moment of this study. The main aim of present research was to develop a method that could, potentially, be practically applicable for industrial potato peel waste recycling into encapsulated phenolic compounds (fine powder), with a further approbation as an antioxidant for ground pork meat. Potato peel wastes were collected from the local potato processing facility, homogenized in the solvent media, and two accelerated extraction technologies (microwave assisted (MAE) and ultrasound accelerated extractions) were applied for the extraction of biologically active compounds and encapsulation wall material. Produced extracts were concentrated (recovered solvent had been collected and reused) and directed for spray-drying. In general, MAE alone showed higher extraction yields than in combinations with ultrasound treatment. Extracts reached maximal biologically active compound concentrations (and were possessing highest radical scavenging activities) after 10 min of MAE treatment. Produced capsules (food grade antioxidant) inhibited ground pork meat lipid oxidation during the storage study at accelerated oxidation conditions. Acquired results form a basis for development of a potato peel industrial scale processing technology.

**Key words:** extraction, encapsulation, oxidation inhibition, potato peel recycling.

### **INTRODUCTION**

According to FOASTAT (2018), the potato production reached 475.9 million tonnes worldwide in 2016. In general, potato consumption vary highly between countries with different cultures and income levels. In lower income countries potatoes are mostly sold raw, while in the First world countries up to 70% of all produced potatoes can be utilized for industrial processing (Parr et al., 2018). In combination with the fact, that up to 40% of total potato mass that enters the production facility can exit it in a form of waste (Schieber et al., 2001) and that the most international waste regulations are commanding to reduce the amount of generated wastes and maximise the recycling and re-use, lately, potato by-product recycling has become a very important topic in the food industry.

As an inexpensive by-product, potato peels contain extractable biologically valuable compounds that can be applied in food and pharmaceutical industries (Al-Weshahy & Rao, 2012; Friedman et al., 2017) and, upon consumption, are able to

provide beneficial effect on the human health (Samarin et al., 2012; Hsieh et al., 2016). Additionally, a potato peel extract can be applied as an antioxidant for various food products prolonging the shelf-life (Habebullah et al., 2010; Mohdaly et al., 2010; Farvin et al., 2012; Samarin et al., 2012). Although, biologically active compound extracts are usually unstable due to their reactivity and require an additional protection/stabilization. One of the possible protection technologies is encapsulation – a method of entrapping targeted compounds in the protective shell or matrix.

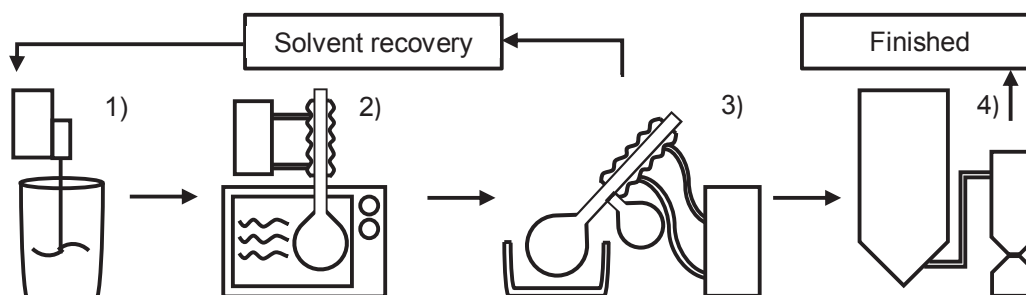
The main aim of present research was to combine both extraction and encapsulation technologies to develop a compact method for encapsulated phenolic compound production (fine powder) from actual industrial potato peel wastes, that, potentially, could be applicable on potato processing sites.

## MATERIALS AND METHODS

### Potato peel waste processing schematics

Industrial potato peel wastes were processed into a food grade antioxidant (fine powder) through following steps (Fig. 1):

- 1) potato peel homogenization in the solvent media;
- 2) valuable compound extraction through microwave assisted extraction and ultrasound treatment;
- 3) solvent evaporation and extract concentration;
- 4) encapsulation of extracted compounds via spray-drying.



**Figure 1.** Steps for the simultaneous phenolic compound extraction and encapsulation. Where, 1) – homogenisation; 2) – microwave assisted extraction; 3) – vacuum evaporation (concentration); 4) – encapsulation through spray-drying.

All steps are described in detail in further sections.

### Chemicals and materials

All reagents and standards (96% ethanol, Folin–Ciocalteu reagent, sodium carbonate, ethyl ether, gallic acid, hexane, isopropanol, isooctane, acetic acid, trolox, 2,2–diphenyl–1–picrylhydrazyl radical (DPPH<sup>•</sup>)) were purchased from the Merck KGaA (previous Sigma–Aldrich).

Industrial potato peel samples (brown skin,  $3 \pm 2$  cm) were acquired from the Paplata Nr. 1, Ltd. (Raubeni, Latvia). Freshly prepared ground pork meat was purchased

from the local market chain Maxima Group (Jelgava, Latvia) and was used on the same day, within two hours after the purchase.

### **Sample preparation**

Industrial potato peel by-products were collected directly from the potato processing lines into the plastic bags. Within an hour, peels were rapidly frozen in the laboratory scale plate freezer FT34-MKII (Armfield, United Kingdom) and stored at  $-20\text{ }^{\circ}\text{C}$  for further analyses.

For extraction purposes, frozen potato peels (25 g) were homogenised in the 60% ethanol (v/v; 250 mL) with a kitchen type blender RHB450-S-W (Rotex, China) for  $30 \pm 1$  s. Acquired samples were evenly divided between three 300 mL Erlenmeyer flasks and used for the further extraction purposes.

### **Simultaneous phenolic compound and carbohydrate extraction**

Microwave EMS 2840 (Electrolux, Sweden) had been used to conduct the microwave assisted extraction (MAE) and three extraction methods had been applied:

- 1) MAE only;
- 2) MAE with following 5 min ultrasound treatment (UST) in the laboratory scale ultrasonic bath YJ5120-1 (Zhengzhou Henan, China);
- 3) 5 min UST with following MAE.

For all three methods, four MAE regimes were applied: 5, 10, 15, and 20 min at the power level of 180 W. This power level had been chosen based on Singh et al. (2011) work reporting that lower power levels result in the higher bioactive compound yield from potato peels.

### **Concentration of extracts**

After the initial extraction, still hot (to minimize sedimentation of dissolved carbohydrates) supernatant had been collected and centrifuged at  $2,500 \times g$  for residue separation in the CM-6MT (Elmi, Latvia) centrifuge. Resulting mixture of phenolic compounds and soluble carbohydrates (with possible impurities of soluble fibre, free amino acids, etc.) was collected and concentrated under the vacuum at  $40\text{ }^{\circ}\text{C}$ . Recovered ethanol was collected and its concentration was adjusted to 60% (based on density) and reused for the further extractions.

### **Encapsulation via spray-drying**

After the concentration, phenolic extract had been directly fed into the bench top type spray-drier B-290 (Buchi, Switzerland) that was operating at the following conditions: inlet and outlet air temperatures were maintained at  $170 \pm 2\text{ }^{\circ}\text{C}$  and  $70 \pm 2\text{ }^{\circ}\text{C}$ ; feed rate:  $15\text{ mL min}^{-1}$ ; spray gas flow:  $667\text{ L h}^{-1}$ . Collected samples were transferred into the glass jar, that was additionally sealed with a paraffin paper and stored in the plastic bag at  $-20\text{ }^{\circ}\text{C}$  till the further application.

### **Determination of moisture content**

Moisture content had been determined by a gravimetric method by the oven drying at  $105\text{ }^{\circ}\text{C}$  to a constant weight, ISO 1442:1997 (iso.org).

### **Determination of the total phenolic compound content in the produced capsules**

Produced capsules were divided in two batches:

1. For the total phenolic compound analysis: 0.1 g of capsules was dissolved in the 2 mL of water and 40 mL of ethanol had been added for carbohydrate sedimentation and phenolic compound dilution. Mixture was placed in the ultrasonic bath for 10 min, centrifuged for 5 min at  $2,500 \times g$  and filtered.

2. For the phenolic compound content analysis on the capsule surface. 0.1 g of capsules were washed with 10 mL of ethanol, centrifuged for 5 min at  $2,500 \times g$  and filtered.

All filtrates were collected for the further analyses.

Jung et al. (2011) method with slight modifications had been applied for the phenolic compound analysis. Briefly, 200  $\mu\text{L}$  of the phenolic extract was mixed with 1 mL of distilled water and 100  $\mu\text{L}$  of Folin–Ciocalteu phenol reagent (precisely diluted with distilled water 1:1, v/v). After 5 min incubation, 2 mL of the 10% sodium carbonate solution was added and mixture was allowed to stand at room temperature for 60 min, and then centrifuged at  $2,500 \times g$  for 10 min. The absorbance was measured at 725 nm by a laboratory spectrophotometer Jenway 6300 (Stone, United Kingdom). Results were recalculated per dry matter and expressed as a gallic acid equivalent (GAE).

### **Determination of the free radical scavenging activity**

DPPH<sup>•</sup> was used for *in vitro* determination of free radical scavenging activity (Yu et al., 2003). Briefly, 0.5 mL of extract was mixed with 0.004% DPPH<sup>•</sup> ethanol solution, and mixture was allowed to stand for 30 min in the dark at room temperature. The absorbance was measured at 517 nm. Data had been expressed as a trolox equivalents per 100 g of sample ( $\text{mg } 100 \text{ g}^{-1} \text{ TE}$ ).

### **Determination of the total lipid content**

The total lipid content was determined by the Soxhlet extraction method AOAC 920.39 Fat (Crude) or Ether Extract in Animal Feed.

### **Ground meat treatment with encapsulated phenolic compounds**

Produced capsules (4.6 g) were added to the ground pork meat (500 g) in proportion 2 g of phenolic compounds per 1 kg of pork fat. Acquired meat mass was homogenized in the kitchen type blender for 30 s and stored at the accelerated oxidation conditions ( $+30 \pm 1 \text{ }^\circ\text{C}$ ), with daily sampling. All experiment had been conducted in triplicate.

### **Lipid extraction from the pork meat mass**

Lipid extraction had been conducted according to the Hara & Radin (1978) method, with modifications. Briefly, meat sample (ca. 15 g) had been mixed with the extraction solution (hexane–isopropanol, 3:2 v/v) in proportion 1 : 6 (w/v). Extraction had been conducted for 30 min under the constant energetic shaking. Supernatant was removed by filtration, and evaporated under the vacuum at  $40 \text{ }^\circ\text{C}$ . Collected condensed solvent was re-used for the extraction of the same sample two more times. Extracted lipids were kept under the vacuum till further analysis.

### **Determination of lipid oxidation ratios**

An active oxygen value was determined according to the International Fragrance Association Analytical Method: Determination of the Peroxide Value (from October 17<sup>th</sup>, 2011) (IFRA, 2011), that is based on the ISO-Standard 3960 third edition 2001, AOCS CD 8b-90, European Pharmacopeia, and Leatherhead Food RA second edition.

### **Microscopic analysis**

Microscope Leica DM300 LED (Leica Microsystems, Germany) had been used for the microscopic analysis. Photos were taken with camera Leica DFC 290 HD and analysed using software Leica Application Systems (LAS) V4.2.

### **Statistical analysis**

Data statistical analysis was conducted in the MS Excel 2016 software. One-way ANOVA ( $P \leq 0.05$ ) and Tukey's test were applied for the statistical analysis of all suitable data clusters. Where applicable, data is shown as a mean value with a standard deviation.

## **RESULTS AND DISCUSSION**

In the present research, a new potato peel (an industrial potato processing by-product) recycling method had been developed. It is scalable and, potentially, practically applicable and consists of several stages (Fig. 1) that include: collected peel homogenisation in the solvent media, initial phenolic compound and carbohydrate extraction, concentration of acquired extracts (recovery of the solvent for further reuse), and following encapsulation through spray-drying. The proposed method offers a basis for actual industrial scale technology development.

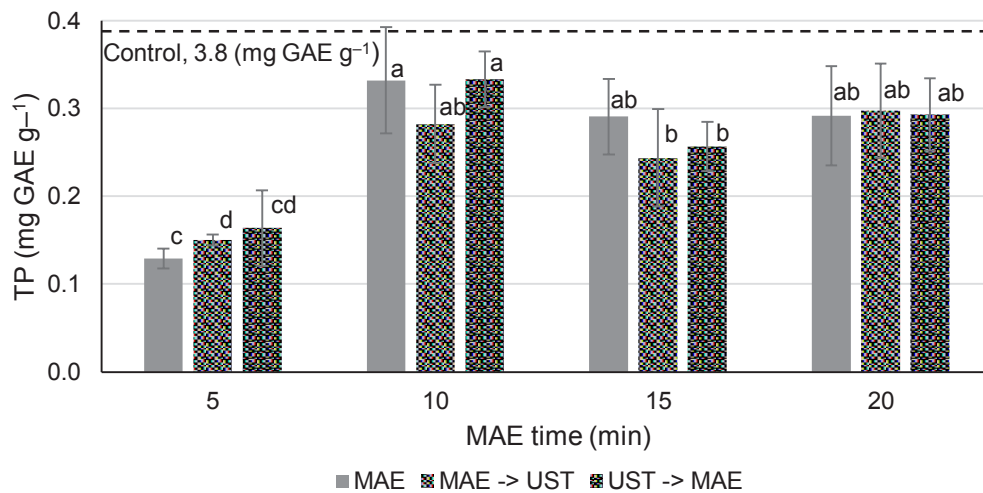
The first step (peel homogenisation) included also the first initial phenolic compound extraction – a conventional extraction, when phenolic compounds (a result of cell disruption and diffusion processes) are released into the solvent. Additionally, in the case of present research (when applied samples were previously frozen) peel samples were defrosted during the homogenisation process. On the industrial production scale, depending on the available machinery and amounts of reprocessible material, it could be necessary to increase homogenisation times in order to achieve complete cell disruption and to rise phenolic compound extraction yields. Our previous research (Shepelev et al., 2016) showed that, for the biologically active compound extraction purposes, abrasion peeled industrial potato peels can be stored for up to two days at ambient conditions (as it is on the actual potato processing plants). In addition, it had been emphasized that it is critical to reprocess only peels with remaining whole cells, but completely shredded peels are not suitable for extraction purposes even directly after the peeling.

The second processing stage included the main extraction step. To achieve shorter extraction times and, at the same time, maximize phenolic compound yields at minimal energy consumption, two accelerated extraction technologies were applied (microwave assisted and ultrasound accelerated extractions) in different combinations. Results show (Fig. 2) that MAE without any additional ultrasound treatment is a method of choice for phenolic compound extraction from ground potato peels. In case of the present research, maximal phenolic compound yield had been achieved after the 10 min long extraction,

and the prolongation of extraction time did not show a statistically significant increase in phenolic compound concentrations in acquired extracts. As had been stated previously, parameters could vary depending on the applied equipment and simultaneously processed amount of peels.

The extraction time of 5 min gave the lowest phenolic compound yield in final extracts. Samples extracted at 15 showed lower results comparing to 10 min, but higher than samples extracted during the 5 min period. The incorporation of ultrasound treatment gave a positive result only in 5 min samples. It can be concluded that MAE allow to achieve maximal molecule agitation and diffusion during the extraction process that makes ultrasound treatment unnecessary. In opposite case, when MAE treatment is at too low rates, ultrasound treatment can increase biologically active compound extraction yields till some extent.

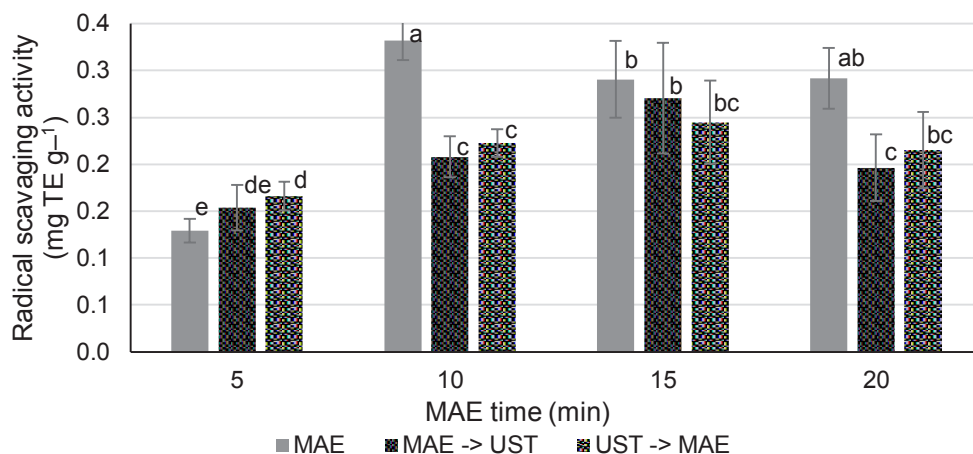
It is known that concentrated ethanol solutions exhibit great antimicrobial activity (Oh & Marshall, 1993). Taking in consideration the applied MAE, it can be concluded that potato peels will undergo complete sterilization during the manufacturing time and a possible microbial contamination during peel storage should not cause problems or trigger necessity of implying additional critical control points. Although, it is advised to minimize or prevent unnecessary pathogenic microflora development as it can result in the presence of stable toxins in produced extracts.



**Figure 2.** Total phenolic (TP) compound extraction yield depending on the extraction method ( $n = 9$ ), similar lowercase letters indicate no significant difference among samples of the same fraction ( $P \leq 0.05$ ). Where, TP – total phenolics; GAE – gallic acid equivalent; MAE – microwave assisted extraction; MAE → UST – microwave assisted extracting with following 5 min of ultrasound treatment; UST → MAE – 5 min of ultrasound treatment with the following microwave–assisted extraction.

Similar results were acquired during free radical scavenging activity analyses (Fig. 3). But in this case, free radical scavenging activity of samples that were extracted for 10 min (and 20 min) dropped significantly with the incorporation of ultrasound treatment. This fact could suggest that completely extracted phenolic compounds could

undergo degradation, as ultrasound treatment was combined with higher temperatures (a result of MAE). An advanced research on different extraction method combinations should be performed to give a more precise conclusion. For the purposes of present research, 10 min MAE had been chosen as an optimal method based on the ratio between the phenolic compound yield and energy consumption.



**Figure 3.** Free radical scavenging activity of produced extracts depending on the extraction method ( $n = 9$ ), similar lowercase letters indicate no significant difference among samples of the same fraction ( $P \leq 0.05$ ). Where, TE – Trolox equivalent; MAE – microwave assisted extraction; MAE → UST – microwave assisted extracting followed by 5 min of ultrasound treatment; UST → MAE – 5 min of ultrasound treatment followed by microwave-assisted extraction.

The third processing stage consisted of the concentration of produced extracts. It had been designed to achieve two main goals: 1) to recover the solvent for repeated extraction (the concentration of ethanol had been adjusted based on density), and 2) to decrease the volume of samples to minimize energy and time consumption for the next stage – encapsulation via spray-drying.

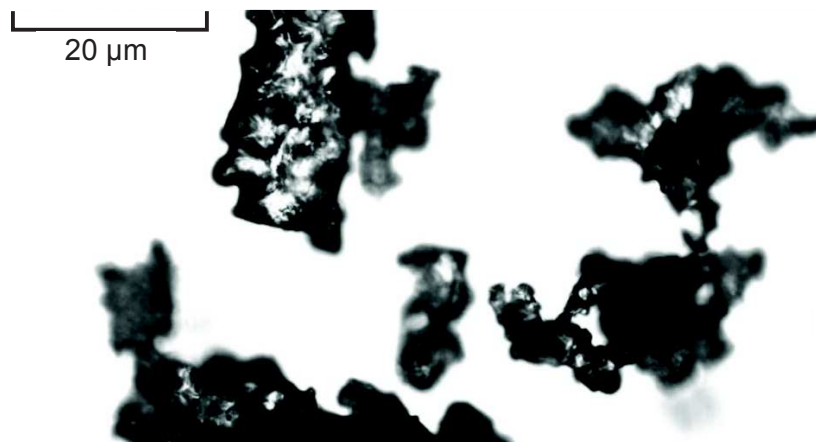
Additional energy savings were achieved by the transfer of hot extracts (directly after MAE) into the vacuum evaporator (at the average extract temperature of  $79.0 \pm 2.8$  °C, upon leaving the MAE chamber). This allowed to achieve a rapid solvent evaporation mostly without additional thermal treatment in the warm water bath. To minimize the possible oxidation, after the concentration, extracts were kept under the constant vacuum until they had been fed into the spray-dryer. It had been previously reported that phenolic extracts can undergo also polymerisation processes resulting in the extract colour change from lighter to darker (Vámos-Vigyázó, 1981).

The final fifth processing stage included the encapsulation of extracted phenolic compounds by using carbohydrate compounds that should be dissolved in the same extract. The comparison of total phenolic compound concentrations and gravimetric data on dry weight of extracts (data is not shown) shows that carbohydrate-to-phenolic mass ratios were ca. 11 : 1 (w/w), which was sufficient for encapsulation purposes.



It had been planned that multiple types of encapsulation could take place during the capsule formation. First of all, formation of the matrix type capsule should occur, as both core and wall materials are water soluble and should form homogeneous structures. Low molecular weight sugars should form a solid shell wall that should prevent oxygen diffusion inside the capsule and active compound oxidation. Secondly, it had been reported that amylose can encapsulate small guest molecules by forming inclusion complexes (Cohen et al., 2008; Putseys et al., 2010; Gökmen et al., 2011; Kong & Ziegler, 2014).

While it was possible to dry all extracts, unfortunately, particles of the dried powder coagulated in the collection chamber, resulting in uneven capsule shapes (Fig. 4). Visual inspection of the transparent collection chamber during the spray-drying process showed that capsules were in the powdered form, and were moving around the chamber due to the strong aeration (construction flaw/compromise of the small laboratory scale benchtop dryer). This fact indicates that the same air that had been used for moisture evaporation and transfer is coming into contact with the dry product in cooled conditions of the collection chamber. As a result, it is possible that moisture could condensate on the particle surface, causing the coagulation. Additionally, active aeration could accelerate encapsulated compound oxidation (Anantharamkrishnan & Reineccius, 2017). Taking in consideration that carbohydrate content in the extract should consist mostly of short chain carbohydrates (as applied 60% ethanol solution as a solvent for phenolic compound extraction should result in the high molecular weight carbohydrate sedimentation), it is possible that temperature in the collection chamber exceeded the glass transition temperature of capsule matrix carbohydrates and resulted in the carbohydrate transition into rubbery state forming the sticky surface. Increased surface area of produced particles (in comparison to a theoretically possible spherical shape capsules) could provide a practical benefit in a way of faster capsule dissolution in target media and more rapid release of encapsulated compounds. Additional trials are necessary in spray-dryers of advanced construction or industrial scale spray-dryers.



**Figure 4.** Irregular shape of produced capsules under the optical microscope.

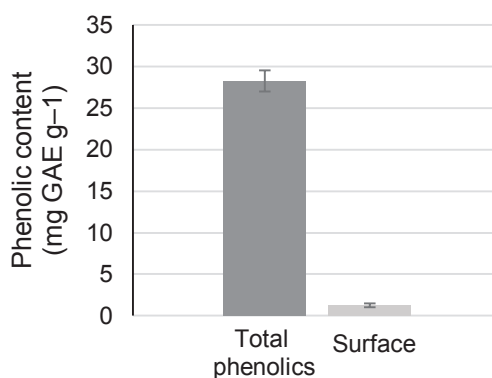


Based on the phenolic compound distribution between capsule surface and total phenolic content (Fig. 5), encapsulation efficiency of produced capsules (the ration between surface and bound compounds) reached  $95.5 \pm 0.9\%$ . This value should be considered as too big, because capsule had been formed of the same polarity substances. As a result, homogenous matrix should be formed and encapsulation efficiency should be a function of surface-to-volume ratio, and surface area should be increased together with the decrease of capsule size. As an example, in our previous study on gallic acid encapsulation in low dextrose equivalent starch showed encapsulation efficiency of  $77 \pm 7\%$  for spray dried samples (Sepelevs et al., 2018). There could be couple of reasons for a such high number. First of all, phenolic compounds could be mechanically separated from the surface during the encapsulation process in the cyclone and withdrawn from the dryer together with the airflow. On the opposite side, as it had been discussed previously, carbohydrates could undergo the glass transition state and (forming sticky rubbery capsule surface) entrap phenolic compounds strong enough preventing their extraction with a pure ethanol (as it was performed in present study, as carbohydrates are not dissolvable in it). Deeper study of the capsule surface is necessary for more precise conclusions.

There are several approaches that could be implemented to improve upon the current product. First of all, assuming that there is a large proportion of low molecular weight carbohydrates in sample extracts, a low dextrose equivalent (4–10) maltodextrin could be added to the concentrated extract before feeding it into the spray-dryer in order to increase the average dextrose equivalent and thus rise the glass transition temperature. Secondly, different extraction solvent combinations with lower ethanol contents could be used to increase solubility of longer chain dextrans. But it is important to take into the consideration that decreased ethanol concentrations could negatively influence phenolic compound extraction yields (Wu et al., 2012).

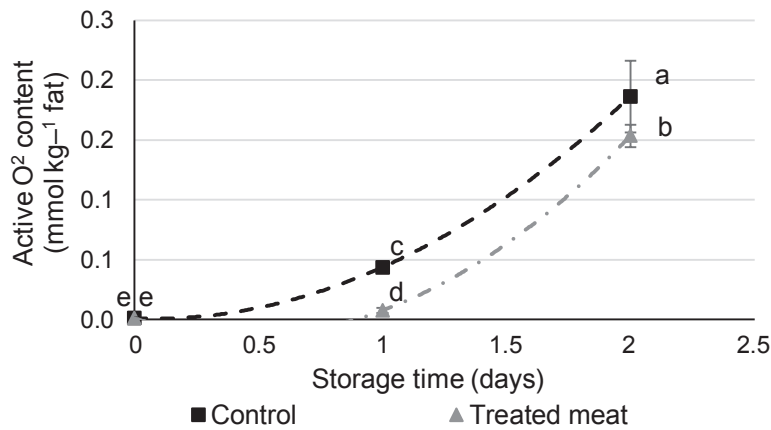
All five stages of the developed method took a half of a day in laboratory conditions, with most of the time spent on the solvent evaporation and recovery. It had been planned that on the production scale all process should take approximately one work day, based on the available concentration technology. Due to the chosen MAE and concentration stages, the present method is suitable for the production process that consists of separate batches. This is a great choice for small scale production lines that could be used in Latvia, due to the relatively small annual potato processing amounts countrywide.

The functionality of produced encapsulated phenolic compounds as a food grade antioxidant had been tested during the ground pork meat storage at accelerated oxidation conditions (Fig. 6). It can be seen that after one day of storage at  $30\text{ }^{\circ}\text{C}$ , meat mass with added encapsulated phenolic compounds shows ca. five times lower oxidation rates in



**Figure 5.** Total and surface phenolic contents of produced capsules (n = 3).

comparison to control (active oxygen contents of 0.08 mmol kg<sup>-1</sup> for treated meat samples versus 0.44 mmol kg<sup>-1</sup> for control samples, respectively). Unfortunately, on the second day of storage oxidation rates of both samples were significantly increased. On the third day of the storage, microbial degradation of meat samples did not allow quantitative determination of active oxygen. The rapid ground pork meat spoilage had been anticipated but a decision had been made against application of additional preservatives as they could interfere with oxidation processes. Results can be evaluated as a positive as present research part had been conducted at increased temperatures.



**Figure 6.** Changes in the active oxygen content in the ground pork meat lipid fraction during the storage at accelerated oxidation conditions ( $n = 3$ ), similar lowercase letters indicate no significant difference among samples of the same fraction ( $P \leq 0.05$ ).

Number of studies reported a successful application of herbal extract as natural antioxidants in meat and meat products (Nissen et al., 2004; Devatkal et al., 2012; Mathenjwa et al., 2012; Ozvural & Vural, 2012; Sánchez-Muniz et al., 2012; Cao et al., 2013; Reddy et al., 2013; Naveena et al., 2013; Grāmatiņa et al., 2017). Although, in majority of those cases, investigators chose raw materials with high phenolic compound contents, as grape skin, green tea leaves, red peony, etc. And only couple of investigators were using actual potato peel extracts, but prepared from potatoes that were peeled in laboratory conditions (Mansour & Khalil, 2000; Kanatt et al., 2005; Farvin et al., 2012). One of the major differences between potato peel samples acquired in laboratory conditions and acquired from the actual potato processing facility is that on the production sites peel by-products usually are being collected with an assistance of water, and, as had been stated previously, water is a second food-grade solvent for phenolic compound extraction after ethanol. Of course, an additional assistance (mechanical, ultrasound, microwave, etc.) should be applied for better phenolic compound diffusion from cells into water, but still some phenolic compound losses should be present. This means that previously reported data is not fully scalable for industrial application, as water and ethanol extracts results in the different phenolic profiles and different antioxidant properties (Farvin et al., 2012).

Theoretically, there could be additional applications of recovered and encapsulated phenolic compounds – introduction of produced capsules as a separate specialized wall material for another valuable compound encapsulation, to ensure the protection of encapsulated materials against oxidation. For example, it could be used for unsaturated oil encapsulation where (incorporated in the modified starch shell wall material) phenolic compounds could serve as an additional oxygen barrier. Additionally, it had been reported that some of water soluble polysaccharides (no specification reported) from potato peels have good water holding abilities, fat binding capacity, foaming properties (due to the presence of some protein remains), emulsification stability, and possess a natural antioxidant abilities (Jeddou et al., 2016). As a result, it can be concluded that the produced mixture of simultaneously extracted phenolic compounds and carbohydrates have a potential to serve as a wall material, for example, for the pharmacological use where active compound preservation plays a critical role and application of more expensive wall materials (and technologies) is a common practice.

## CONCLUSION

A practically applicable method for the industrial potato peel waste processing and powdered encapsulated phenolic compound production had been developed, using the industrial potato peel by-products as the only source material. It consists of four subsequent steps: peel homogenisation in the solvent media, phenolic compound and carbohydrate extraction, produced extract concentration, and encapsulation via spray-drying. Microwave assisted extraction alone showed higher extraction yields than in combinations with ultrasound treatment. Produced capsules showed a positive result during the ground pork meat accelerated storage study during the first two days of the storage at accelerated oxidation conditions. The developed method does not produce chemical wastes that could be toxic for the environment. Acquired results form a basis for development of a potato peel industrial processing technology.

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