Valorization of agro-industrial byproducts: Extraction and analytical characterization of valuable compounds for potential edible active packaging formulation

Food Packaging and Shelf Life

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Abstract:	Large amounts of byproducts having high environmental impact are produced in the agro-industrial field. However, they are often rich of bioactive molecules and can represent a good source of new products. In a sustainability context, supporting Circular Economy project, this work aimed at extracting active compounds from onion, artichoke and thistle byproducts, and exploited new applications in the field of food packaging. Compound characterization achieved by chromatographic techniques demonstrated the presence of valuable ingredients such as quercetin and chlorogenic acid, and prebiotic carbohydrates. A high content of total phenolic compounds was evaluated mainly in artichoke and onion byproducts, and the addition of the extracts to oil samples showed a significant increase of oxidative stability, measured by Oxitest, even higher than 100% respect to blank oil. An active film based on the use of edible substances has been realized, and preliminary data about its application on food products showed promising developments.
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Response to Reviewers:	

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DI PARMA

DIPARTIMENTO DI SCIENZE
CHIMICHE, DELLA VITA E DELLA
SOSTENIBILITÀ AMBIENTALE

Parma, 2022-04-14

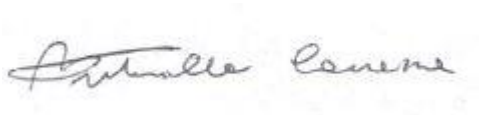
Dear Editor,

we attach the files of our new revision of the manuscript entitled “Valorization of agro-industrial byproducts: extraction and analytical characterization of valuable compounds for potential edible active packaging formulation” by Maria Grimaldi, Antonella Cavazza, Olimpia Pitirollo, Paola Ornaghi, Claudio Corradini.

We have responded point by point to all the requests received from the reviewers, and modified the text following their suggestion. We think that the quality of the present version of the manuscript is improved, and hope that this text will be considered suitable for publication on your Journal.

Sincerely,

Antonella Cavazza



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Response to reviewers

Please find enclosed the list of comments followed by our response in red character.

The manuscript has been modified accordingly to the suggestions, using red colour.

Reviewer #1: Comments on manuscript FPSL-D-21-00863, entitled "Valorization of agro-industrial byproducts: extraction and analytical characterization of valuable compounds for active packaging formulation" by Grimaldi et al.
The paper is interesting with the both approaches on active compound extract and evaluation of their activity and applications in edible films, but not so original. The paper is well written, but need some improvements to be accepted as described hereafter. Therefore, I suggest major revision.

Detailed comments :
Title ; the title should be more explicite aiming at the edibility, thus should specify "edible active packaging formulation"

R. We modified the title as suggested, taking also into account the suggestion of reviewer n. 2 (adding the word "potential")

Line 69=L69, please, refer to the regulation related to natural compound and food contact material ability.

R. References to regulation have been added to the revised text, as also requested by reviewer 2.

L95 : please specify if coatings or self-standing films are concerned, and if active coatings have been applied on support films to activate the surface.

R. Articles cited in line 95 are about self-standing films based on edible ingredients. Other studies such as those cited in line 68 (Lantano, Corradini) are about coatings applied on support films, but are not edible. The text has been modified to convey these informations.

L96 as edibility is concerned, the paper should be focused only toward coatings and not self-standing films that are not aimed to be edible.

R. The sentence regards the article from Atares and Chiralt, dealing with both edible films and coatings. Self-standing films are proposed as biodegradable material that can be removed before food consumption, but can be even eaten with the product. The text has been slightly modified to underline the importance of biodegradability.

L115 what did you meant by "secondary packaging"? like cardboard, or over "primary packaging" ?
R. We were referring to cardboard, and modified the text accordingly, to specify it.

L135 why 75°C, it is not conventional procédure for dry mass determination of food samples. How long was the drying, or was the duration adapted until constant dry weight ?

R. The drying conditions were adapted with the aim of limiting the thermal stress and protect thermolabile compounds. For this reason temperature was set at lower value than concentional. The duration was prolonged until constant weight. Those informations have been added to the text.

L144 is it refers to dry sample, I think it is not the dry weight, but dried vegetable at 75°C. It would be more excat to say dried vegetable than dry weight which should follows the standardized procedure for dry mass or water content determination

R. The sentence has been modified as suggested

L144 "falcon" ?? it is a bird ! Did you meant vial or flask ?

R. We replaced the term with “vial”

L159-212 as the chromatography techniques have been set up in previous paper, please in parts 2.5 and 2.6 and 2.7, refer to the published papers.

R. we cited previous papers in section 2.5 and 2.6, as similar chromatographic conditions were reported in previous works, although the methods were slightly modified. Conditions of section 2.7 were not previously published.

L245 what was the role of σD-(+)-gluconic acid-lactone in the medium acidification. Was the pH controlled of measured?

R. gluconic acid-lactone was used according to the cited article. We did not investigate the effect on pH, although this molecule dissociates producing a proton and it is used as acidifier. The pH value was measured, and the recorded value was about 5. This information has been added to the revised text.

L253 how was chosen the ratio extracts/solution ? What will be the dry matter ratio reported to the film ?

R: The extracts/solution ratio was chosen based on preliminary results from the analysis of oxidative capacity performed by Oxitest. The revised text was implemented with this information.

L256 please explain why these RH-T°C conditions selected for film conditioning prior measurements? Std refers either to 38°C-90% or 25°C-50%.

R. We selected those conditions according to previous experiments performed in our lab in the late years. We didn’t perform a real optimisation, but have found, also in accord with the article by Barbut and Harper (2019) that room temperature and high humidity values improved film transparency. The text has been revised to convey this information and the reference was added.

L260-269 were the RH and T° controlled during mechanical tests ? Were samples kept contact at 80%RH ?

R. Temperature and RH were not controlled during the test.

L273-276 how the food samples packed in films ? Are they wrapped, in sealed paoches, or coated with wet solution of the film forming coatings ? Please, add a picture of packed/or coated samples as supplémentary data.

R. We specified in the text that food samples were wrapped in the films (not coated with wet solution). A picture of meat samples has been added to supplementary data (Figure 3S).

L308-339 part 3.2 : only a qualitative analysis of carbohydrates has been done. Is it possible to quantify the amount in each extract ?

R. We performed quantitative analysis for analytes whose standards are commercially available. Table 1 reports data of the examined extracts.

L398-402 the antioxidant activity (induction time) should be normalized or related to the estimated content in polyphenols.
Please discussed results in that way more in detail or give the curve discussed in lines 408-409.

R. We added a figure to Supplementary materials (Figure 2S) reporting the curve showing correlation between polyphenols amount and IP values. Text was revised accordingly.

L414 I wonder where is the coherence between biodegradability approach and edibility approach of the packaging. In my mind, the objectives are very different, between edibility which requirements are very more struct and biodegradability. Of course, if it is edible, it is biodegradable, but the application and end of life are not same. I believe you should focus only on edible property!

R. We modified the sentence according to reviewer's suggestion, pointing out that the packaging can be eaten with the product. Of course it depends on the product: alginate films covering hamburger can be easily eaten, and the enrichment with aromatic substances can even improve organoleptic properties of the meat. For other products such as strawberries the impact on the organoleptic properties would be probably too strong.

L424-427 I don't understand why plasticization was discussed, as all films contained same amount of glycerol. However, the TS and EB changes should be discussed according the phenolic compounds. A review focuses on the effect of polyphenols incorporation on the mechanical and barrier properties of edible films (cf Benbettaieb N. et al. Bioactive edible films for food applications: influence of the bioactive compounds on film structure and properties. Critical Reviews in Food Science and Nutrition, 2017, 597, 1137-1153 doi.org/10.1080/10408398.2017.1393384)

Same comment for discussion lines 428-433, the change should be discussed according the extract incorporation, and possible interactions with plasticizer of interactions between alginate strings..

R: The text has been modified avoiding to discuss about plasticizers, but only referring to the parameters affecting film features. The text has been enlarged pointing out the effect of incorporation of polyphenols on mechanical properties. A reference to the suggested review has been inserted. The discussion about calcium ions has also been implemented referring to the extract incorporation and possible interactions.

L446 where is it shown? picture 4 is not so clear. Was a piece of film apply on the surface of the beef hamburger? How long time the test conducted?

R. The revised text has been modified underlining that Figure 4 reports some different films, and also shows an example of beef hamburger covered by an edible film. We also added information about test conditions to section 2.10.

L451-459, please quantify the assumptions/statements! How was the oxidation reduced (test used and magnitude of decrease), how long the shelf-life prolonged, etc.. please give picture of mould growth on uncoated beef hamburger compared to wrapped ones etc..
This part should state clearly it was preliminary tests, to be confirmed by quantitative analysis and measurement of beef quality.
R. the text has been rewritten according to the suggestions. It has been specified that only a limited and preliminary evaluation based on visual exam was conducted, and that a punctual quantitative analysis should be performed to prove such data. Some more information about samples aspect have been provided.

Mould growth was observed on fruits controls, and a related picture has been reported in Supplementary files (Figure 4S).

Figure 3 : please improve quality, give legend between upper and lower graph, translate Italian (presione) into English…

R. The figure has been revised as requested, and legend has been implemented.

Figure 4 , please add legend to better identifiy the differences between the films on the upper part of the picture, same comments for beef hamburger and film deposit on its top.

R. Letters have been inserted on the figure to better identify the films shown. Figure caption was accordingly enriched to help the reader.

Reviewer #2: The work is focused on the extraction and chromatographic characterization of bioactive molecules from agro-food waste and byproducts in a circular economy perspective, and their potential application as antioxidants in biodegradable films for food packaging.

Considering the main topics of the journal, the authors should have studied more in depth the part

relating to the characterizations on packaging materials, for example by conducting migration tests of the extracts loaded into the polymer, through the use of regulated food simulants to study the release kinetics rather than to report a food contact test without specific characterizations other than visual changes. For now, apart from the mechanical tests, the results of which have not been reported in detail, no other analyzes have been conducted to assess the effectiveness of these materials for use as packaging. However, the authors have specified that this is preliminary work. Therefore, considering the current scientific interest towards the recovery of substances with high added value from agro-food waste, and considering the substantial work carried out by the authors in terms of characterization of different types of extracts from plant byproducts, this paper can certainly contribute to the advancement of scientific research in the field of food packaging.

It is suggested to change the title of the work as follows: "Valorization of agro-industrial byproducts: extraction and analytical characterization of valuable compounds for potential active packaging formulation".

R. We modified the title as suggested, taking also into account the suggestion of reviewer n. 1 (adding the word “edible”)

It would be useful for the authors to provide a graphical abstract highlighting the experimental steps of the work.

R. A graphical abstract has been provided

English could be improved.

R. We have rewritten some sentences trying to improve the language form.

In the introduction, authors should specify the chosen extraction technique and justify the reason by reporting a comparison with other extraction techniques, including bibliographic references.

R: a brief paragraph has been added to the introduction, discussing the choice of extraction techniques. A reference about comparison of different extraction methods has been inserted.

Line 69: Authors have to add the reference to the European Regulation referred to (European Commission Regulation No. 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food, Official Journal of the European Union, 12, 75-80).

R: reference has been added

Globally, the bibliography should be updated by citing more recent articles. Here are some suggestions:
line 45: doi.org/10.1016/j.biortech.2020.122755;
line 58: doi.org/10.1016/j.biortech.2021.124684;
line 61: doi.org/10.1016/j.fpsl.2019.100396, doi.org/10.1016/j.porgcoat.2019.105487;

line 67: doi:10.3390/foods9111628;
line 82-84: 10.1002/ceat.202100044, 10.3303/CET2187098;
line 97: doi.org/10.1016/j.fpsl.2021.100756, doi.org/10.1016/j.lwt.2018.05.049;

R: more recent references have been inserted, including some of those suggested.

Here are some writing errors to correct:
Line 163: add space between 8 and um and add a comma after um;
Line 164: remove spaces between mL and min as in the rest of the text;
Line 167: in KDa, the K goes in lowercase;
Line 174: add space after 25;
Line 178: add spaces between 2 x 250;
Line 180: add space after 125;
Line 192: X in lowercase. Remove spaces between mL and min;
Line 210: "e" in quercetin is in red, please adjust;
Line 222: remove space in ° C;
Line 244: adjust spaces between 0.300 and g/g;
Line 245: adjust spaces in the brackets;
Line 374: add a dot after leaves.

R. Thank you, we corrected the writing errors

Line 437: the authors could add SEM images to confirm this sentence.

R. Unfortunately SEM is not available in our department. We only performed some image acquisitions using a microscope, but the picture did not show interesting information

Is the aluminum disc on which the solutions were cast fixed or moving? What size is it?

R. The disc was fixed, and information on size (30 cm^2) has been added.

Did the authors check the thickness uniformity of the resulting films? If so, how? Add this information in section 2.10.

R. Tickness was measured by means of a micrometer on seven different small regions of 3 cm^2 each. The information has been added to section 2.10.

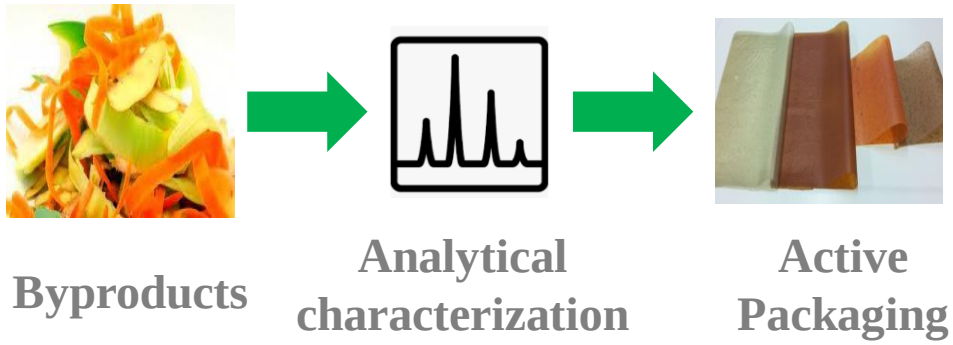
In addition, mechanical test results should be added, either in tabular or graphical form (stress-strain curves) and compared with other biopolymers. Although it was not the main objective of the work, the mechanical tests conducted were the only form of characterization of the packaging films, therefore the results obtained with statistical analysis should be reported.

R. A Table reporting data related to mechanical test (Table 3) has been added. Statistical analysis was also provided. The text has been enriched with a brief discussion and comparison with literature data.

Highlights

- Bioactive compounds were extracted by onion, artichoke and thistle byproducts
- Chromatographic analyses provided identification of antioxidants and prebiotics
- Extracts were incorporated in edible active films proposed as packaging
- Application of films showed promising results in reducing food deterioration

Graphical Abstract



Valorization of agro-industrial byproducts: extraction and analytical characterization of valuable compounds for potential edible active packaging formulation

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Abstract

Large amounts of byproducts having high environmental impact are produced in the agro-industrial field. However, they are often rich of bioactive molecules and can represent a good source of new products. In a sustainability context, supporting Circular Economy project, this work aimed at extracting active compounds from onion, artichoke and thistle byproducts, and exploited new applications in the field of food packaging. Compound characterization achieved by chromatographic techniques demonstrated the presence of valuable ingredients such as quercetin and chlorogenic acid, and prebiotic carbohydrates. A high content of total phenolic compounds was evaluated mainly in artichoke and onion byproducts, and the addition of the extracts to oil samples showed a significant increase of oxidative stability, measured by Oxitest, even higher than 100% respect to blank oil. An active film based on the use of edible substances has been realized, and preliminary data about its application on food products showed promising developments.

Keywords: active packaging; agro-industrial byproducts; edible film; biopolymer; phenolics; prebiotics.

1. Introduction

In the late years, world politics is moving towards a more sustainable economy, implementing the ambitious Circular Economy plan with the aim of reducing waste and reusing byproducts. The main goal would be to introduce wastes in the productive cycle again, to obtain new resources that can be used as new raw materials, providing an additional economic value (Scarlet, 2013).

In details, a great amount of agricultural products is not considered suitable for commercial large scale distribution because of not adequate dimension, shape, maturation stage; besides abundant materials such as stems and leaves are discarded along industrial processes and during technological transformation, thus requiring the need of waste disposal at very high cost.

Artichokes, onions, and thistles are examples of plants largely cultivated in many districts of Italy. Many factories process them in order to produce canned and frozen food; therefore huge amounts of byproducts are obtained during technological treatments, and can be considered a source of interesting compounds such as polyphenols and prebiotics (Ameer, Shahbaz, & Kwon, 2017).

Such products are still rich in bioactive compounds, and non-edible parts rather often contain even a higher amount of such substances than the edible portions, therefore, they can represent a good source of new useful products, in the context of the sustainability theme, and accordingly to the concept of circular economy (Sherwood, 2020). In fact, growing attention is paid to reach the goal of a Zero Waste Economy, where wastes are inserted again into the productive cycle, becoming a new resource (Sharma et al., 2021).

The potential of natural extracts is well known since long time (K. Wang, Lim, Tong, & Thian, 2019). Traditionally, spices and herbs were widely used for their antioxidant and antibacterial properties to protect food from spoilage (Cavazza, Corti, Mancinelli, Bignardi, & Corradini, 2015), and also in many other fields of application such as food technology, cosmetics, nutrition and biotechnology, and lately in the field of active packaging (Bhardwaj, Alam, & Talwar, 2019; Wrona et al., 2021).

Active packaging adds a new role to the traditional functions attributed to packaging, as it is aimed not only at containing and protecting the product from mechanical and physical stress, but has also the purpose of interacting with it by transferring active molecules, and has the effect of prolonging its stability. Many examples of active coatings applied on support films can be found in literature (Claudio Corradini, Alfieri, et al., 2013; Lantano et al., 2014).

The use of natural ingredients is a very important strategy to gain costumer acceptance, and to accomplish the strict regulation concerning food contact materials (Commission Regulation (EU) No 10/2011, 2004, 2011; Commission Regulation (EU) No 450/2009, 2009). In the late years, a great attention has been focused to the use of plastic, and to control the possible migrations of additives to the product. Indeed, the presence of non-intentionally added substances (NIAS) and emerging contaminants not included in the regulation has been reported by many researchers (Bignardi, Cavazza, Laganà, Salvadeo, & Corradini, 2017; Gómez Ramos, Lozano, & Fernández-Alba, 2019; Ubeda, Aznar, Rosenmai, Vinggaard, & Nerín, 2020), and new analytical methods have been implemented for their characterization (Bignardi, Cavazza, Laganà, Salvadeo, & Corradini, 2018). Besides, waste management of packaging materials poses a serious concern from the ecological point of view, and a great attention to sustainability has been raised over the past years.

In order to reduce risks of contamination from plastic materials and following the recent European regulation underlining the necessity to limit plastic use, the choice of natural substances to obtain innovative materials for food packaging represents a good solution. Many examples of biodegradable packaging obtained avoiding the use of petroleum derivatives, and based for example on starch and natural extracts, are reported in recent literature studies (Khalid et al., 2018; Medina-Jaramillo, Ochoa-Yepes, Bernal, & Famá, 2017; Seligra, Medina Jaramillo, Famá, & Goyanes, 2016).

Some authors proposed biodegradable packaging solutions realized from waste material such as raw material from crops (Davis & Song, 2006), seafood waste (de la Caba et al., 2019), mango peels (Adilah, Jamilah, Noranizan, & Hanani, 2018), or carrot wastes (Otoni et al., 2018). An important feature is the functionalization of packaging with active ingredients that confer added properties such

92 as antioxidant activity, as in the case of a film based on blueberry fibers (de Moraes Crizel, Haas
93 Costa, de Oliveira Rios, & Hickmann Flôres, 2016), or the durian leaf extract in gelatin-based films
94 (Joanne Kam et al., 2018). The effect of the addition of natural extracts such as essential oils for
95 antibacterial and antioxidant properties to improve the oxidative stability has been also demonstrated
96 (Atarés & Chiralt, 2016; Cavazza et al., 2015). Some previous studies related to the development of
97 self-standing films reported use of ginger oil (Maria et al., 2016) and plant extracts (Bonilla & Sobral,
98 2016) such as green tea (Li, Miao, Wu, Chen, & Zhang, 2014). An important prerogative of some of
99 these bio-films is their biodegradability, but also their edibility, since they are realized starting from
100 natural and edible ingredients such chitosan, gelatin, alginate (Atarés & Chiralt, 2016).
101 On these basis, the hypothesis of the present work was to demonstrate if a usually discarded material,
102 that is generally considered and treated as a waste, can become, after being submitted to extraction
103 and characterized in terms of chemical composition, a source of valuable products. Its use could be
104 proposed in the field of packaging, and exploited to obtain active edible films, with the goal to protect
105 food from deterioration. This objective would perfectly fit in the Circular Economy and sustainability
106 context.
107 The work was organized in separate steps, starting from the set-up of an extraction procedure
108 involving agroindustrial artichoke, onions, and thistle byproducts, followed by the analytical
109 characterization of the substances by HPLC, and the measurement of the phenolic compounds
110 content. Different extraction methods comparing conventional and innovative techniques for
111 maximizing bioactive compounds recovery from vegetable matrices have been explored (Catena et
112 al., 2020). In particular, in the late years, much attention is paid to green methods having limited
113 effect on the environment, thus conventional extraction techniques are being often replaced with new
114 technological processes such as the assisted ultrasound extraction (AUE) that limits the amount of
115 needed solvent, and allows time reduction.
116 The addition of extracts to vegetable oil was tested to evaluate the effect exerted on its oxidative
117 stability, measured by means an Oxitest reactor, used to accelerate oxidation process with high

temperature and oxygen pressure. A potential use of the extract was the realization of an active and edible packaging enriched with functional ingredients, choosing alginate as substrate for its ability to form a biopolymer when reacting with calcium ions (Gholamian, Nourani, & Bakhshi, 2021). The obtained packaging could be even classified as edible film, and its enrichment with onions, rich of active molecules such as prebiotics (Otoni et al., 2018), represents a potential innovative strategy to enrich a food product, also providing functional features. Finally, to reach the zero-waste goal, the residue after extraction can be proposed as a bulk material for secondary packaging, for example cardboard production.

2. Materials and methods

2.1 Chemicals

PTFE (Polytetrafluoroethylene) 0.2 µm x 25 mm filters, Nylon 0.2 µm x 25 mm filters (Agilent Technologies, Milan, Italy), water (MilliQ), ethanol 96% , methanol 99%, acetone, sodium carbonate, Folin-Ciocalteu reagent, gallic acid, sodium alginate, calcium carbonate, sodium acetate, sodium hydroxide 50% w/w, glycerol, D-(+)-gluconic acid-σ-lactone (GDL) theobromine, quercetin, quercitrin, chlorogenic acid, glucose, fructose, sucrose, 1-kestose, nystose, fructofuranosyl-nystose, dextrans analytical standards were purchased from Sigma Aldrich (Steinheim, Germany).

2.2 Samples and treatment

Byproducts from artichokes (*Cynara Scolymus*), red onions (*Allium Cepa* var. Tropeana), and thistles (*Cirsium vulgare*) were obtained from Italian agro-food industries. Aliquots of each vegetable were processed by mixing together all vegetable parts (leaves, stems, parts of flowers etc.). Moreover, in order to explore the possible differences in total phenolic content, and oxidative stability of the different parts of the vegetables, other sample aliquots were obtained by separating leaves of artichokes, onions and thistles from their respective stems. The different vegetable parts were

processed separately, as described in the following sections. Dry weight was determined on each sample by drying aliquots of 10 grams in an oven set at 75 °C, to protect thermolabile compounds, until constant weight was reached.

2.3 Extraction procedure for Folin-Ciocalteu assay and HPLC-UV-DAD-MS analysis (phenolic extracts)

The material was submitted to extraction after a preliminary quick homogenization using liquid nitrogen. The conditions selected for the experiments were based on a previous work (Rinaldi et al., 2020), with some modifications, consisting in particular, in the use of ultrasound assisted extraction (UAE) (Dai & Mumper, 2010). Different solvents such as water, ethanol and acetone were tested. The final procedure selected to perform the experiments was as follows: sample amounts corresponding to 0.5 g of dried vegetable were placed in a 50 mL vial; then, 20 mL of acetone were added, and submitted to UAE in a beaker covered with aluminum foil. Subsequently, the samples were placed in a centrifuge at 4000 g for 10 minutes, and supernatant was recovered and placed in a 100 mL flask. To achieve an exhaustive extraction, the procedure was repeated for three consecutive times, and aliquots of solvent were collected and brought to dryness. The extracts were then recovered with 15 mL of ethanol and filtered through a PTFE filter before analysis.

2.4 Extraction procedure for Steric Exclusion Chromatography and HPAEC-PAD analysis

Aliquots of byproducts samples were dried in an oven at 65 ° C for 72 h and then minced. Aqueous extracts were obtained by suspending 0.25 g of sample in 25 mL of (MilliQ) water, and treated at 80 °C for 60 minutes under stirring, whereas alcoholic extracts were prepared using 25 mL ethanol at 50 °C for 60 minutes. Then, samples were centrifuged at 4000 g for 30 minutes at 4 °C and the supernatant was taken up and diluted with (MilliQ) water 1: 5 and filtered through a Nylon 0.2 µm x 25 mm filter (Claudio Corradini, Lantano, & Cavazza, 2013).

169 **2.5 Characterisation of extracted compounds by Steric Exclusion Chromatography**

170 The analysis of the composition of the extracts, in terms of high molecular weight fractions
171 corresponding to the polysaccharide fraction, was assessed through the use of an Agilent 1200 series
172 HPLC (Milan, Italy) equipped with an Agilent 1260 Infinity refractive index detector. The column
173 employed was a steric exclusion column PL aquagel-OH40 (8 µm, 300 x 7.5 mm) thermostated at 30
174 °C. The elution was performed using water containing 0.2% sodium azide, at a flow of 0.5 mL/min
175 in isocratic mode. The injection volume was 100 microliters (N. González-Ballesteros et al., 2019).
176 All samples were injected in triplicate after 1:35 dilution. To obtain references of the molecular
177 weights, standards of dextrans having variable molecular weight from 150 to 8 kDa, were previously
178 injected. The choice of these molecules over other available standards, such as polyethylene oxides,
179 is linked to the fact that the structure of the dextrans can be considered similar to that of the natural
180 polysaccharides occurring in the byproducts.

181

182 **2.6 Analysis of carbohydrates by HPAEC-PAD**

183 Carbohydrates separations were performed using a DX500 series Dionex liquid chromatograph
184 (Sunnyvale, CA, USA), equipped with an AS50 Dionex autosampler with a 25 µL loop, and an ED50
185 model pulsed electrochemical detector (PAD) with Ag / AgCl reference electrode and gold working
186 electrode. A degassing module allowed to keep eluents under constant flow of helium. The software
187 CHROMELEON was used for recording chromatographic data.

188 Fructans analyses were conducted by using a CarboPac column PA 100 (2 x 250 mm), at room
189 temperature, and a flow rate of 1 mL/min. A linear gradient was performed with an increasing
190 concentration of sodium acetate from 0 to 125 mM in 90 minutes, followed by column washing and
191 initial conditioning (C. Corradini et al., 2004). Glucose, fructose, sucrose, 1-kestose, nystose,
192 fructofuranosyl-nystose were the commercially available standards used to identify peaks by
193 comparison of retention times. A calibration curve was constructed for the above mentioned

194 standards, using five concentration levels from 5 µg/mL to 100 µg/mL. All measurements were
195 performed in triplicate.
196
197 **2.7 Analysis of phenolic compounds by HPLC-DAD-MS**
198 Phenolic compounds were characterized by reversed phase chromatography on an Agilent 1200 liquid
199 chromatograph equipped with binary pump, degasser, autosampler, column thermostat and diode
200 series detector (DAD). The system was coupled by an electrospray (ESI) interface to linear ion trap
201 analyzer mass spectrometer (MSD Trap XCT Ultra, Agilent Technologies). The chromatographic
202 separation of the analytes was carried out by means of a Phenomenex, Luna C18 (2) 100Å 5 µm, (250
203 x 2 mm). Column flow was set at 0.500 mL/min. The elution was performed following a programmed
204 gradient with two different mobile phases, A (H_2O / HCOOH 95:5) and B (acetonitrile / HCOOH
205 95:5). The elution program was the following: from 0 to 4 minutes, % B increased from 0.1 to 12.5
206 and held until 7 minutes; from 7 to 8 minutes % B was raised up to 20 and held until 13 minutes; at
207 14 minutes % B was set at 60 until 19 minutes; at 20 minutes % B increased to 100, holding for 7
208 minutes. Finally, the column was reconditioned for 5 minutes.
209 UV-DAD detection was carried out at a wavelength of 280 nm. For mass spectrometry detection,
210 electrospray ionization was set in positive mode (ESI +). The nebulizer gas (nitrogen, 99.9% purity)
211 and the dry gas (nitrogen, 99.9% purity) were delivered at pressure of 60.0 psi and 10.0 L/min,
212 respectively. Other conditions of the interface were: capillary voltage 4.5 kV, skimmer voltage 40.0
213 V, drying gas temperature 365 °C. Masses were recorded in SIM (Selected Ion Monitoring) mode,
214 selecting signals of molecular ions $[M+H]^+$ at m/z 303 for quercetin, m/z 449 for quercitrin, m/z 355
215 for chlorogenic acid and m/z 181 for theobromine used as internal standard. Confirmation of
216 compounds identity was performed by a comparison of migration time and mass spectrum obtained
217 from sample and standard solutions. Agilent ChemStation B.01.03 and LC–MSD Trap 6.0 Build
218 458.0 softwares were used for instrument control and data processing.

Quantitative analysis was performed by means of mass spectrometry. Since no white matrix was available to build a blank calibration curve, the quantitative analysis was performed using the method of standard additions. Four concentration levels were built for chlorogenic acid, quercetin and quercitrin from 12.5 to 100 µg/mL. Theobromine was used as internal standard at fixed concentration of 5 µg/mL.

2.8 Total Phenolic Content (TPC)

UV-vis spectrophotometric analyses (by a Thermo Scientific™ Evolution™ 201/220, Milan, Italy) were performed to determine the total polyphenolic content (TPC) following the Folin-Ciocalteu (FC) method, based on the addition of an oxidizing reagent that, in the presence of phenolic residues, causes a change in the absorbance of the solution. The reaction is based on phosphotungstate and phosphomolybdate that, in a basic environment, oxidizes the -OH group to a carbonyl group reducing itself and giving a blue color to the solution (Ammor & Jennan, 2019).

50 µl of sample were mixed with 1160 µl of water (MilliQ), 300 µl of sodium carbonate 20% w/w, and 100 µl of the Folin-Ciocalteu reagent; the solution was then introduced into an oven at 40 °C for 30 minutes. An identical preparation was performed on blank solvent. Absorbance was measured at 760 nm. The TPC value was expressed as µg of GAE (gallic acid equivalent)/ g of dry sample by means of a calibration curve built in the range between 1 and 30 µg/mL of gallic acid. All measures were performed in triplicate.

2.9 Analysis by Oxitest

Oxitest reactor (VELP Scientifica, Usmate, Italy) allowed the evaluation of the oxidative stability of a model matrix, sunflower oil, enriched with the phenolics extracts of thistle, onion and artichoke by-products. The stability was determined by accelerating the oxidation process of 10 g of sample using high temperatures (90 °C) and oxygen pressure at 6 bar. A sample of sunflower oil was chosen as reference. The induction value of the same sample enriched with plant extract derived from 0.5 g (dry

245 weight) was evaluated. In order to minimize the volume differences between blank and enriched
246 samples, the extracts were preventively dried in a rotary evaporator and resuspended using the model
247 oil. Measures were performed in duplicate employing two chambers of the reactor. The software
248 allows to follow the oxidative process inside two reaction chambers, and graphically displays the
249 recorded oxidation curve, calculating the induction period (IP).
250
251 **2.10 Active and edible films**
252 Active edible films were formulated on the basis of a previous research (Benavides, Villalobos-
253 carvajal, & Reyes, 2012) according to the following procedure. A 1% w/v solution of sodium alginate
254 was prepared adding the salt in distilled water at 70 °C under vigorous stirring until complete
255 solubilization. Glycerol was added in an amount of 0.300 g/g of sodium alginate. In a different beaker,
256 a suspension of $CaCO_3$ (0.04 g/g of alginate) and GDL (5.4 g/g of $CaCO_3$) was prepared in 100 mL
257 of distilled water. The pH value of the solution was monitored, and recorded value was about 5.0.
258 The suspension was then added to the alginate solution under vigorous stirring (100 g), kept at 55 °C
259 for 30 minutes to allow the removal of CO_2 from the reaction environment, and placed in an ultrasonic
260 bath for 40 minutes in order to eliminate bubbles. Bubbles were formed as a result of both the
261 homogenization and the release of Ca^{++} ions promoted by GDL with the related development CO_2;
262 this last point is very important since CO_2 may be responsible for cavities formation in the gel during
263 the cross-linking phase, thus compromising the final result (Benavides et al., 2012).
264 To obtain the film, 15 mL of byproduct phenolic extracts (from artichoke, or onion, or thistle) were
265 added to 150 mL of solution under moderate stirring (20 g) for 70 minutes. The amount of extract to
266 add to the solution was selected based on preliminary results evaluating the antioxidant effect by
267 Oxitest, in order to obtain a significant effect. 55 mL of the final mixture was then poured into
268 aluminum disks of 30 cm^2, and placed for 18 hours at 50 °C. Blank films, obtained without extract
269 addition were also formulated. The films were left in a climatic chamber at 22 °C and 80% humidity
270 for 12 hours, to allow the conformational rearrangement of the polymer chains before use. The high

level of humidity was selected since it was found to improve film transparency, in accord to preliminary experiments and to Barbut and Harper (Barbut & Harper, 2019).

Thickness uniformity of the resulting film was checked by measuring seven different regions of 3 cm^2 each by means of a micrometer.

Characterization of film properties, such as tensile strength (TS), and percentage elongation at break (EB%) were performed.

TS and EB% were evaluated using a Micro Stable System texture analyzer (Spinea, Venezia, Italy). TS was performed according to ASTM Method D882 (ASTM, 2012a). The film was cut into 10 cm × 1.5 cm rectangles, the initial grip separation was set to 50 mm, and the cross-head speed was set to 0.2 mm/min. Data have been processed by software EXPONENT. At least five replicates of each film were tested. Tensile strength (TS, MPa) was calculated using the following equation:

$$TS = \frac{F\ max}{Area}$$

where F_{max} is the maximum force needed to break the sample (N), and A is the cross-sectional area

The elongation at break was calculated as the percentage increase in sample length. Deformed length (L) and original length (L0) were calculated the EB% as following equation:

$$EB\ \% = \frac{L - L0}{L0} * 100$$

Where L is the elongation at the moment of rupture (m), and L_0 is the initial length or grip length of the sample (m).

Some preliminary experiments on food storage were conducted by using the active film for package of meat (see Supplementary data), cheese, and fruits such as strawberries and cranberries. Three equal aliquots of each food products were wrapped in films, and then stored at refrigerating conditions (4-6 °C), or at room temperature: one was packaged with the active film; another one was used as control and packaged with a blank film (with no extract); a third one was covered with an aluminum foil. The products were examined at time intervals of 12 hours for four consecutive days, by checking the main

296 organoleptic properties, such as color, smell, presence of mould, and surface roughness in terms of
297 freshness appearance.
298
299 **2.11 Statistical analysis**
300 SPSS statistical software (Version 19.0, SPSS Inc., Chicago, Illinois, USA) was used to calculate
301 means and standard deviations (SD), and to verify significant differences between samples by one-
302 way analysis of variance (ANOVA) followed by Tukey's honest significant difference (HSD) test at
303 $p \leq 0.05$ to identify differences among groups.
304
305 # 3. Results and discussion
306
307 **3.1 Analysis by steric exclusion chromatography**
308 Aqueous and alcoholic extracts of byproducts (constituted by a mixture of different vegetable parts)
309 were submitted to analysis by steric exclusion chromatography. The presence of molecules, probably
310 belonging to polysaccharides family have been evidenced in all samples. The chromatograms
311 obtained (see supplementary materials) from respectively a thistle aqueous, and alcoholic extracts
312 showed the presence of a main band eluting at 23 minutes, that in the aqueous medium is accompanied
313 by a smaller one eluting at about 20 minutes. The comparison with retention time of dextran standards
314 permitted the attribution of a molecular weight to these fractions. In details, the main signal at 23
315 minutes represents molecules smaller than 8000 Da, whereas the band at 20 minutes can be ascribed
316 to compounds having a mass of approximately 8000 Da. Some traces of compounds of higher
317 dimension could also be noticed in the aqueous extract by the presence of three signals located at
318 lower retention time (around 11.0, 12.5 and 17.3 minutes).
319 The analysis of artichoke and onion byproducts showed similar profiles, with small differences from
320 the quantitative point of view.

About a possible attribution of the identity of the substances, previous studies on bioactive components of artichoke, onion and thistle byproducts have revealed that they are rich in phytochemicals. Many of these compounds are characterised by molecular weights compatible with the reported results, such as phenols, and carbohydrates as fructans (Cavazza et al., 2010).

3.2 Analysis of carbohydrates

Conventional techniques for the determination of sugars are not useful for separating mixtures of all carbohydrates. The use of the strong anion exchange chromatography system (HPAEC) and its combination with pulsed amperometric detection (PAD), characterized by high sensitive and selective detection, provides a really efficient separation tool, and has been successfully used in many fields (Claudio Corradini, Lantano, et al., 2013; Lee, 1996). It allows to separate multicomponent mixtures containing monosaccharides, disaccharides, oligosaccharides, and even sugar alcohols in the same run, also giving the possibility of separating anomeric and positional isomers (Borromei et al., 2010). Extracts from residues of thistle, onions and artichokes (containing a mixture of the different vegetable parts) were found to contain simple sugars and also presence of fructooligosaccharides. Those molecules are characterized by prebiotic activity and possess important technological features for their properties to form gel when mixed with water. This phenomenon can be exploited to obtain bulk effect and to improve texture of food preparations such as ice-creams, yogurt, sweets etc. (Claudio Corradini, Lantano, et al., 2013).

From the chromatogram shown in Figure 1, related to an onion extract, it can be seen that many peaks are eluted, showing the presence of sugars of different degree of polymerization.

Available standards were used to identify, on the basis of their retention times, peaks corresponding to the monosaccharides glucose and fructose, the disaccharide sucrose, and molecules with higher degree of polymerization such as 1-kestose, 1-nystose, and 1F-1-β-D-fructofuranosylnystose. Other peaks eluting at longer retention times were tentatively attributed to oligosaccharides 1F-(1-β-D-fructofuranosyl)-2-nystose, 1F-(1-β-D-fructofuranosyl)-3-nystose, and the inulooligosaccharides

(IOS) inulobiose, inulotriose, inulotetraose, inulopentaose, inulohexaose, and inuloheptaose. The assignment was performed referring to the generally accepted assumption that the retention time of a homologous series of carbohydrates increased as the degree of polymerization (DP) increases (Borromei et al., 2010).

Quantitative analysis was performed to determine the amount of glucose, fructose, sucrose, 1-kestose, nystose and fructosylnystose, by referring to calibration curves built with standard solutions. Table 1 shows the amount of each carbohydrate, and it can be seen that glucose was the most abundant compound, followed by sucrose and fructose. As for oligosaccharides, a consistent amount of kestose was recorded, accompanied by traces of nystose and fructofuranosylnystose.

Analysis of artichoke and thistles byproducts showed a less rich fractions of oligosaccharides, limited to few peaks of low intensity.

3.3 Analysis of phenolic compounds

Extracts were prepared by UAE, an efficient extraction technique able to form bubbles in solvent, collapsing near the surface of a solid matrix, and penetrating cell walls, thus enhancing the diffusion of phenolics (Dai & Mumper, 2010; J. Wang, Sun, Cao, Tian, & Li, 2008). Different solvents such as ethanol, acetone and a mixture of water/ethanol 1:1 were tested to evaluate the yield. Previous works reported that each solvent can be considered effective to extract a different class of compounds: for example, anthocyanins that are polar molecules were mainly extracted by methanol, whereas acetone was the most effective for the other compounds (A. A. De Araújo et al., 2014; Lourenço, Moldão-Martins, & Alves, 2019; Pradal, Vauchel, Decossin, Dhulster, & Dimitrov, 2016). Accordingly, our results, based on TPC spectrophotometric analyses, showed higher values when acetone was used.

Artichoke, thistle and onion leafs and stems were found to be rich in polyphenolic compounds. The main molecules occurring were chlorogenic acid in artichoke, eluting at about four minutes, and quercetin in onion, eluting at 6.5 minutes; its glycosidic form, quercitrin, eluting at 3 minutes, was

373 also recorded. The identity of all compounds was confirmed by comparison of retention time and
374 mass spectra with those obtained by standard analysis.
375 Since no white matrix was available to build a blank calibration curve, the quantitative analysis was
376 performed using the method of standard additions. The recorded data (see Table 1) showed that in
377 artichoke leaves and thistle the most abundant compound occurring was chlorogenic acid, followed
378 by quercitrin, and small quantities of quercetin. In onion, quercitrin has been identified as the most
379 abundant, followed by quercetin, while chlorogenic acid was not detected. These results are in
380 accordance with literature data (Alarcón-Flores, Romero-González, Martínez Vidal, & Garrido
381 Frenich, 2014).
382 Presence of caffeic acid was also evaluated, but it was found only in stems. This molecule is a
383 precursor of chlorogenic acid, and the reason for its presence in stems and not in leaves can be linked
384 to the distinct accumulation of metabolites in the different vegetable tissues (Fratianni, Tucci, Palma,
385 Pepe, & Nazzaro, 2007; Sánchez-Rabaneda et al., 2003).

386 The amount of total phenolic compounds was measured by the Folin Ciocalteu assay. Results are
387 showed in Figure 2, where the comparison between samples, demonstrated by statistical evaluation,
388 points the attention to the different content of phenolics found in the examined vegetables. Definitely
389 lower contents were found in both thistle stems and leaves comparing to artichoke and onions.
390 Furthermore, a significant difference is observed also in the different parts of the plant, as can be seen
391 for onion and artichoke stems and leaves. The value recorded for artichokes leaves was approximately
392 four times higher than that of artichoke stems, and comparable to onion stems, whereas the latter
393 shows a value about 1.5 times higher than onion leaves. These results are in accord with Lombardo
394 et al., and Ben Rejeb et al. (Lombardo, Pandino, Ierna, & Mauromicale, 2012; Rejeb, Dhen, Gargouri,
395 & Boulila, 2020). The great difference observed between onion and artichoke or thistle stems can be
396 ascribed to the completely different structure related to the type of the stem tissue, that in onion is
397 soft and similar to its bulb, whereas in artichoke and thistle is more ligneous.

Considering the opportunity of extracting bioactive molecules from byproducts, besides the yield, it has to be also taken into account the very large amount of available raw material: around 95 million tons of onions and more than one million tons of artichokes are grown all over the world. For the latter, only a portion corresponding to 40% is edible, whereas the 60% is considered waste.

3.4 Evaluation of oxidative stability of oil enriched with extracts

The chemical reactions that occur between oxygen and some sensitive components of food represent one of the most important causes of qualitative alteration of the products. In order to test the antioxidant power of extracts, the residues were dissolved in a model product such as sunflower oil, and the oxidative stability conferred to it was evaluated by Oxitest. This instrument has been successfully employed to measure the induction period, which is directly linked to the resistance to oxidation of a product (Cavazza et al., 2015).

Data obtained measuring the induction periods evidenced an increase of the stability of all enriched samples, respect to sunflower oil alone. Figure 3 reports an example of the graphs recorded for a blank oil (A), respect to oil enriched with onion byproducts (B). In the graphs, the oxygen pressure trend during time is reported, and the vertical line indicates the flex point in correspondence to the oxygen consumption linked to the oxidation starting point.

Table 2 shows all values of induction period recorded. A definite higher oxidative stability can be observed for all samples respect to the control. These results demonstrate the positive effect of extract addition in preventing the oil degradation. In details, induction time of sunflower oil was about 500 minutes, considerably lower than that of all examined extracts, whereas enriched samples values ranged from a minimum of 616 ± 22 minutes (corresponding to an increase of about 20%), recorded in the experiment with thistle samples, to a maximum of 1037 ± 49 minutes recorded for the artichoke sample (increase of 107%). These data demonstrated that a strong effect on vegetable oil protection is exerted by the extracts, and suggest a potential use of those materials for preventing oils degradation

423 during storage, such it is done when preparing aromatized oils with onion, rosemary or chilli pepper
424 (Cavazza et al., 2015).
425 To evaluate whether the effect on oil stability was linked to the amount of phenolic compounds
426 occurring in the extract, the correlation between gallic acid equivalents measured by Folin-Ciocolteu
427 assay, and IP values obtained by Oxitest measures was considered. A linear trend was observed (see
428 Figure 2S, Supplementary Data), and suggested that phenolic compounds can be considered
429 responsible for the activity of vegetable oil stabilization.
430
431 **3.5 Alginate films enriched with extracts**
432 As shown by previous literature studies (Lourenço et al., 2019), different kind of edible films with
433 naturally antioxidant compounds have been developed to extend the shelf-life of fresh foods, offering
434 a material able to replace plastic packaging, and that can be even eaten with the product. In some
435 foods, such as hamburger, an aromatic film could improve the organoleptic properties, unless the
436 impact on the taste and palatability is too strong.
437 To this aim, the extracts object of this study, in particular those from artichoke and onions, for the
438 abundant presence of polyphenols and prebiotics, respectively, were incorporated in alginate-based
439 films, and a preliminary study on the effects of the composition and the ratio between ingredients on
440 the film properties was conducted.
441 The tensile strength of the films obtained ranged from 83.2 ± 1.2 to 100.9± 0.7 MPa (see Table 3),
442 and significant differences can be noted between blank alginate films and all materials enriched with
443 the different vegetable extracts. In detail, onion films showed the highest value. The %EB also
444 increased from 5 % to 12 % compared to a film based on only alginate. These results are in agreement
445 with literature data (Di Donato et al., 2020a) showing higher values of tensile strength for films
446 embedded of waste vegetable extracts containing oligosaccharides, probably due to the introduction
447 of polar groups that physically interact with alginate, and able to decrease the chain-chain hydrogen
448 bonding. It is known that mechanical and structural properties of a film are strongly dependent by

many parameters such as plasticizer amount, film crosslinking degree, but also by the addition of byproducts extracts (A. Araújo et al., 2018; Di Donato et al., 2020b; Luchese, Spada, & Tessaro, 2017; Mahcene et al., 2020; Piermaria et al., 2011; Straccia, Romano, Oliva, Santagata, & Laurienzo, 2014). Indeed, natural compounds can act as plasticizers or anti-plasticizers, as underlined in the review by Benbettaieb et al. (2019), (Benbettaïeb, Karbowiak, & Debeaufort, 2019). In particular, simple phenolic compounds can be involved in interactions or chemical linkages with groups of the polymer chains, leading to an improvement of the functional properties of the films. The resulting effect can be a reduction of intermolecular forces that increases the mobility of polymer chains, with enhancement of flexibility and extensibility, although an opposite behavior was recorded for polyphenols such as tannins.

Moreover, the effects of metal ions on crosslinking, and consequently on the physical properties of alginate films has to be taken onto account, as natural extracts can interact with cations. For instance, flavonoids can form complexes with metal cations, with possible effects on the mobility of chains. Liling et al. (Liling et al., 2016) investigated the different behavior of cations finding out effects on tensile strength and light transmission, underlining the important role of the media composition on film properties.

Our experiments showed that addition of onion byproducts, containing fructooligosaccharides, in alginate film, gave added value in terms of resistance, and more cohesive structure with respect to films made using artichoke or thistle byproducts. This result can be attributed to higher viscosity and formation of smaller pores, as previously reported by authors performing studies on biopolymer gels containing fructooligosaccharides (Silva & Sato, 2017). As for addition of artichoke byproducts, containing mainly bioactive compounds belonging to polyphenols, an increase of the flexibility was observed. This can be explained by the action of phenolic compounds affecting the morphology and the strength of film matrix. The hydrophilic groups of phenolic compounds increase the interaction among alginate molecules thus contributing to an increased mobility, similarly to that reported by Iskender & Yemenicioğlu for zein films (Arcan & Yemenicioğlu, 2011).

475 Some active and edible films obtained are reported in Figure 4, showing the possibility to modulate
476 some properties such as the color, depending on the ingredient used. Moreover, an example of the
477 application of a film applied on a beef hamburger surface is shown. It is remarkable that film features
478 can be customised in terms of transparency, colour, thickness, texture, flavour, tailoring the aromatic
479 profile according to the type of product to be packaged. This represents a potential innovative strategy
480 to enrich a food product in terms of flavour and palatability, also providing functional properties that
481 can represent an added value to a product.
482 The main application performed, still at a limited preliminary level, regarded experiments on food
483 products storage evaluation. Some investigations were carried out, and showed higher durability and
484 prolonged shelf-life of the samples treated with the active packaging in terms of oxidative stability
485 (Bignardi C.; Carà D.; Cavazza A.; Corradini C.; Grimaldi M.; Salvadeo P., 2016). Preliminary
486 evaluation by a visual exam suggested an increment of shelf-life for meat (See Figure 3S,
487 Supplementary materials) and vegetable products. The main organoleptic properties were considered:
488 colour of treated meat samples was found to be stable for three days, and fruits exhibited a fresh
489 aspect until the fourth day, with significant differences compared to controls. Of course, these data
490 should be confirmed by quantitative analysis, and by more punctual determinations. The enhancement
491 of oxidative stability could be ascribed to the activity of antioxidant compounds such polyphenols
492 occurring in the active film. Moreover, a visual exam of vegetable surfaces permitted to observe
493 mould development only on untreated samples (see Fig 4S, Supplementary Data), although further
494 selective experiments should be performed to evaluate the punctual effect on microorganisms growth.
495 However, even though data recorded about the use of active packaging based on byproducts extract
496 are still limited, they show that it can be a promising tool to reduce food deterioration and act against
497 food waste.
498 Since these films could be proposed as edible material, to be consumed together with the food product,
499 besides their action of preservation, they can improve the flavor and the aroma of products, conferring
500 additional properties to promote the consumption thanks to their positive effects on the palatability.

Moreover, they can be considered a potential ingredient to enhance the nutritional value of a product since, for example, artichoke extract can be considered a source of antioxidant, and onion extract a source of prebiotics.

4 Conclusion

This work was aimed at evaluate if a material that is generally discarded by agroindustrial plants could be valorized and become useful to be re-inserted in a productive cycle. The proposed application regarded the field of packaging, with a main objective of protecting food products from degradation.

The experiments performed on extraction, and on analytical characterization, demonstrated the presence of valuable bioactive substances in all material considered. In particular, artichoke and onion were found to be rich in polyphenols such as chlorogenic acid and quercitrin, and prebiotic oligosaccharides, respectively. Therefore, such material can be considered as a valuable source of molecules having promising features, able to improve oxidative stability of food products, as demonstrated with experiments on sunflower oil, and as evaluated by spectrophotometric assay. Their use has been proposed for active biobased packaging production, or may be used as nutraceutical ingredient or in functional foods, cosmetic, or herbal fields. Besides, residues after extraction, mainly constituted by cellulose and lignin, could be used further, as raw material for obtaining paper to be adopted as secondary packaging, accomplishing the zero-waste goal. Another prospect could be the production of nanocellulose, rather than metallic nanoparticles that can be obtained in presence of residues of polysaccharides and polyphenols (Noelia González-Ballesteros et al., 2019), and have been successfully used to realize effective packaging (Kumar, Shukla, Baul, Mitra, & Halder, 2018).

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References

Adilah, A. N., Jamilah, B., Noranizan, M. A., & Hanani, Z. A. N. (2018). Utilization of mango peel extracts on the biodegradable films for active packaging. *Food Packaging and Shelf Life*. https://doi.org/10.1016/j.fpsl.2018.01.006

Alarcón-Flores, M. I., Romero-González, R., Martínez Vidal, J. L., & Garrido Frenich, A. (2014). Determination of Phenolic Compounds in Artichoke, Garlic and Spinach by Ultra-High-Performance Liquid Chromatography Coupled to Tandem Mass Spectrometry. *Food Analytical Methods*. https://doi.org/10.1007/s12161-014-9852-4

Ameer, K., Shahbaz, H. M., & Kwon, J. H. (2017). Green Extraction Methods for Polyphenols from Plant Matrices and Their Byproducts: A Review. *Comprehensive Reviews in Food Science and Food Safety*. https://doi.org/10.1111/1541-4337.12253

Ammor, K., & Jennan, S. (2019). Total Polyphenol Content and Antioxidant Power of Ammi visnaga from CODEN (USA): PCHHAX Total Polyphenol Content and Antioxidant Power of Ammi visnaga from Morocco, (December).

Araújo, A., Galvão, A., Filho, C. S., Mendes, F., Oliveira, M., Barbosa, F., … Bastos, M. (2018). Okra mucilage and corn starch bio-based film to be applied in food. *Polymer Testing*. https://doi.org/10.1016/j.polymertesting.2018.09.010

Arcan, I., & Yemenicioğlu, A. (2011). Incorporating phenolic compounds opens a new perspective to use zein films as flexible bioactive packaging materials. *Food Research International*. https://doi.org/10.1016/j.foodres.2010.11.034

Atarés, L., & Chiralt, A. (2016). Essential oils as additives in biodegradable films and coatings for active food packaging. *Trends in Food Science and Technology*. https://doi.org/10.1016/j.tifs.2015.12.001

Barbut, S., & Harper, B. A. (2019). Dried Ca-alginate films: Effects of glycerol, relative humidity, soy fibers, and carrageenan. *Lwt*, *103*(January), 260–265. https://doi.org/10.1016/j.lwt.2019.01.004

552 Benavides, S., Villalobos-carvajal, R., & Reyes, J. E. (2012). Physical , mechanical and antibacterial properties
553 of alginate film : Effect of the crosslinking degree and oregano essential oil concentration. *Journal of*
554 *Food Engineering*, *110*(2), 232–239. https://doi.org/10.1016/j.jfoodeng.2011.05.023
555 Benbettaïeb, N., Karbowiak, T., & Debeaufort, F. (2019). Bioactive edible films for food
556 applications:Influence of the bioactive compounds on film structure and properties. *Critical Reviews in*
557 *Food Science and Nutrition*, *59*(7), 1137–1153. https://doi.org/10.1080/10408398.2017.1393384
558 Bhardwaj, A., Alam, T., & Talwar, N. (2019). Recent advances in active packaging of agri-food products : a
559 review. *Journal of Postharvest Technology*, *07*(1), 33–62. Retrieved from
560 http://jpht.info/index.php/jpht/article/viewArticle/20486
561 Bignardi C.; Carà D.; Cavazza A.; Corradini C.; Grimaldi M.; Salvadeo P. (2016). Development and
562 characterization of active alginate-based edible films for food packaging applications. In *book of*
563 *abstract silae 2016*.
564 Bignardi, C., Cavazza, A., Laganà, C., Salvadeo, P., & Corradini, C. (2017). Release of non-intentionally added
565 substances (NIAS) from food contact polycarbonate: Effect of ageing. *Food Control*.
566 https://doi.org/10.1016/j.foodcont.2016.07.013
567 Bignardi, C., Cavazza, A., Laganà, C., Salvadeo, P., & Corradini, C. (2018). Optimization of mass spectrometry
568 acquisition parameters for determination of polycarbonate additives, degradation products, and
569 colorants migrating from food contact materials to chocolate. *Journal of Mass Spectrometry*.
570 https://doi.org/10.1002/jms.4037
571 Bonilla, J., & Sobral, P. J. A. (2016). Investigation of the physicochemical, antimicrobial and antioxidant
572 properties of gelatin-chitosan edible film mixed with plant ethanolic extracts. *Food Bioscience*.
573 https://doi.org/10.1016/j.fbio.2016.07.003
574 Borromei, C., Cavazza, A., Corradini, C., Vatteroni, C., Bazzini, A., Ferrari, R., & Merusi, P. (2010). Validated
575 HPAEC-PAD method for prebiotics determination in synbiotic fermented milks during shelf life. In
576 *Analytical and Bioanalytical Chemistry*. https://doi.org/10.1007/s00216-010-3510-5
577 Catena, S., Rakotomanomana, N., Zunin, P., Boggia, R., Turrini, F., & Chemat, F. (2020). Solubility study and

intensification of extraction of phenolic and anthocyanin compounds from Oryza sativa L. 'Violet Nori.' *Ultrasonics Sonochemistry*, *68*. https://doi.org/10.1016/j.ultsonch.2020.105231

Cavazza, A., Corradini, C., Giannetto, M., Greco, R., Mori, G., & Vicchio, T. (2010). Studio del profilo di fruttooligosaccaridi in cipolle di diversa cultivar mediante HPAEC-PAD: valutazione chemiometrica (pp. 550–553). Marsala (TP).

Cavazza, A., Corti, S., Mancinelli, C., Bignardi, C., & Corradini, C. (2015). Effect of the Addition of Chili Pepper Powder on Vegetable Oils Oxidative Stability. *JAOCS, Journal of the American Oil Chemists' Society*. https://doi.org/10.1007/s11746-015-2738-9

Commission Regulation (EU) No 10/2011. (2004). REGULATION (EC) No 1935/2004 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 27 October 2004 on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC. *Official Journal of the European Union*.

Commission Regulation (EU) No 10/2011. (2011). COMMISSION REGULATION (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food. *Offical Journal of the European Union*.

Commission Regulation (EU) No 450/2009. (2009). COMMISSION REGULATION (EC) No 450/2009 of 29 May 2009 on active and intelligent materials and articles intended to come into contact with food. *Official Journal of the European Union*.

Corradini, C., Bianchi, F., Matteuzzi, D., Amoretti, A., Rossi, M., & Zanoni, S. (2004). High-performance anion-exchange chromatography coupled with pulsed amperometric detection and capillary zone electrophoresis with indirect ultra violet detection as powerful tools to evaluate prebiotic properties of fructooligosaccharides and inulin. *Journal of Chromatography A*, *1054*(1–2), 165–173. https://doi.org/10.1016/j.chroma.2004.07.109

Corradini, Claudio, Alfieri, I., Cavazza, A., Lantano, C., Lorenzi, A., Zucchetto, N., & Montenero, A. (2013). Antimicrobial films containing lysozyme for active packaging obtained by sol-gel technique. *Journal of Food Engineering*, *119*(3), 580–587. https://doi.org/10.1016/j.jfoodeng.2013.05.046

Corradini, Claudio, Lantano, C., & Cavazza, A. (2013). Innovative analytical tools to characterize prebiotic carbohydrates of functional food interest. *Analytical and Bioanalytical Chemistry*. https://doi.org/10.1007/s00216-013-6731-6

Dai, J., & Mumper, R. J. (2010). Plant phenolics: Extraction, analysis and their antioxidant and anticancer properties. *Molecules*. https://doi.org/10.3390/molecules15107313

Davis, G., & Song, J. H. (2006). Biodegradable packaging based on raw materials from crops and their impact on waste management. *Industrial Crops and Products*. https://doi.org/10.1016/j.indcrop.2005.05.004

De Araújo, A. A., Soares, L. A. L., Assunção Ferreira, M. R., De Souza Neto, M. A., Da Silva, G. R., De Araújo, R. F., … De Melo, M. C. N. (2014). Quantification of polyphenols and evaluation of antimicrobial, analgesic and anti-inflammatory activities of aqueous and acetone-water extracts of Libidibia ferrea, Parapiptadenia rigida and Psidium guajava. *Journal of Ethnopharmacology*. https://doi.org/10.1016/j.jep.2014.07.031

de la Caba, K., Guerrero, P., Trung, T. S., Cruz-Romero, M., Kerry, J. P., Fluhr, J., … Newton, R. (2019). From seafood waste to active seafood packaging: An emerging opportunity of the circular economy. *Journal of Cleaner Production*. https://doi.org/10.1016/j.jclepro.2018.09.164

de Moraes Crizel, T., Haas Costa, T. M., de Oliveira Rios, A., & Hickmann Flôres, S. (2016). Valorization of food-grade industrial waste in the obtaining active biodegradable films for packaging. *Industrial Crops and Products*. https://doi.org/10.1016/j.indcrop.2016.04.039

Di Donato, P., Taurisano, V., Poli, A., Gomez d'Ayala, G., Nicolaus, B., Malinconinco, M., & Santagata, G. (2020a). Vegetable wastes derived polysaccharides as natural eco-friendly plasticizers of sodium alginate. *Carbohydrate Polymers*, *229*(September 2019), 115427. https://doi.org/10.1016/j.carbpol.2019.115427

Di Donato, P., Taurisano, V., Poli, A., Gomez d'Ayala, G., Nicolaus, B., Malinconinco, M., & Santagata, G. (2020b). Vegetable wastes derived polysaccharides as natural eco-friendly plasticizers of sodium alginate. *Carbohydrate Polymers*. https://doi.org/10.1016/j.carbpol.2019.115427

630 Fratianni, F., Tucci, M., Palma, M. De, Pepe, R., & Nazzaro, F. (2007). Polyphenolic composition in different
631 parts of some cultivars of globe artichoke (Cynara cardunculus L. var. scolymus (L.) Fiori). *Food*
632 *Chemistry*. https://doi.org/10.1016/j.foodchem.2007.01.044
633 Gholamian, S., Nourani, M., & Bakhshi, N. (2021). Formation and characterization of calcium alginate
634 hydrogel beads filled with cumin seeds essential oil. *Food Chemistry*.
635 https://doi.org/10.1016/j.foodchem.2020.128143
636 Gómez Ramos, M. J., Lozano, A., & Fernández-Alba, A. R. (2019). High-resolution mass spectrometry with
637 data independent acquisition for the comprehensive non-targeted analysis of migrating chemicals
638 coming from multilayer plastic packaging materials used for fruit purée and juice. *Talanta*.
639 https://doi.org/10.1016/j.talanta.2018.08.023
640 González-Ballesteros, N., Diego-González, L., Lastra-Valdor, M., Rodríguez-Argüelles, M. C., Grimaldi, M.,
641 Cavazza, A., … Simón-Vázquez, R. (2019). Immunostimulant and biocompatible gold and silver
642 nanoparticles synthesized using the: Ulva intestinalis L. aqueous extract. *Journal of Materials*
643 *Chemistry B*, *7*(30), 4677–4691. https://doi.org/10.1039/c9tb00215d
644 González-Ballesteros, Noelia, Rodríguez-Argüelles, M. C., Prado-López, S., Lastra, M., Grimaldi, M., Cavazza,
645 A., … Bigi, F. (2019). Macroalgae to nanoparticles: Study of Ulva lactuca L. role in biosynthesis of gold
646 and silver nanoparticles and of their cytotoxicity on colon cancer cell lines. *Materials Science and*
647 *Engineering C*. https://doi.org/10.1016/j.msec.2018.12.066
648 Joanne Kam, W. Y., Mirhosseini, H., Abas, F., Hussain, N., Hedayatnia, S., & Florence Chong, H. L. (2018).
649 Antioxidant activity enhancement of biodegradable film as active packaging utilizing crude extract
650 from durian leaf waste. *Food Control*. https://doi.org/10.1016/j.foodcont.2018.02.036
651 Khalid, S., Yu, L., Feng, M., Meng, L., Bai, Y., Ali, A., … Chen, L. (2018). Development and characterization of
652 biodegradable antimicrobial packaging films based on polycaprolactone, starch and pomegranate rind
653 hybrids. *Food Packaging and Shelf Life*. https://doi.org/10.1016/j.fpsl.2018.08.008
654 Kumar, S., Shukla, A., Baul, P. P., Mitra, A., & Halder, D. (2018). Biodegradable hybrid nanocomposites of
655 chitosan/gelatin and silver nanoparticles for active food packaging applications. *Food Packaging and*

656 *Shelf Life*, *16*(April), 178–184. https://doi.org/10.1016/j.fpsl.2018.03.008
657 Lantano, C., Alfieri, I., Cavazza, A., Corradini, C., Lorenzi, A., Zucchetto, N., & Montenero, A. (2014).
658 Natamycin based sol-gel antimicrobial coatings on polylactic acid films for food packaging. *Food*
659 *Chemistry*, *165*, 342–347. https://doi.org/10.1016/j.foodchem.2014.05.066
660 Lee, Y. C. (1996). Carbohydrate analyses with high-performance anion-exchange chromatography. *Journal*
661 *of Chromatography A*. https://doi.org/10.1016/0021-9673(95)00222-7
662 Li, J. H., Miao, J., Wu, J. L., Chen, S. F., & Zhang, Q. Q. (2014). Preparation and characterization of active
663 gelatin-based films incorporated with natural antioxidants. *Food Hydrocolloids*.
664 https://doi.org/10.1016/j.foodhyd.2013.10.015
665 Liling, G., Di, Z., Jiachao, X., Xin, G., Xiaoting, F., & Qing, Z. (2016). Effects of ionic crosslinking on physical
666 and mechanical properties of alginate mulching films. *Carbohydrate Polymers*.
667 https://doi.org/10.1016/j.carbpol.2015.09.034
668 Lombardo, S., Pandino, G., Ierna, A., & Mauromicale, G. (2012). Variation of polyphenols in a germplasm
669 collection of globe artichoke. *Food Research International*.
670 https://doi.org/10.1016/j.foodres.2011.06.047
671 Lourenço, S. C., Moldão-Martins, M., & Alves, V. D. (2019). Antioxidants of natural plant origins: From
672 sources to food industry applications. *Molecules*, *24*(22), 14–16.
673 https://doi.org/10.3390/molecules24224132
674 Luchese, C. L., Spada, J. C., & Tessaro, I. C. (2017). Starch content affects physicochemical properties of corn
675 and cassava starch-based films. *Industrial Crops and Products*.
676 https://doi.org/10.1016/j.indcrop.2017.09.020
677 Mahcene, Z., Khelil, A., Hasni, S., Akman, P. K., Bozkurt, F., Birech, K., … Tornuk, F. (2020). Development and
678 characterization of sodium alginate based active edible films incorporated with essential oils of some
679 medicinal plants. *International Journal of Biological Macromolecules*.
680 https://doi.org/10.1016/j.ijbiomac.2019.12.093
681 Maria, E., Alexandre, C., Lourenço, R. V., Mônica, A., Barbosa, Q., Cristina, I., … José, P. (2016). Gelatin-

682 based fi lms reinforced with montmorillonite and activated with nanoemulsion of ginger essential oil
683 for food packaging applications. *Food Packaging and Shelf Life*, *10*, 87–96.
684 https://doi.org/10.1016/j.fpsl.2016.10.004
685 Medina-Jaramillo, C., Ochoa-Yepes, O., Bernal, C., & Famá, L. (2017). Active and smart biodegradable
686 packaging based on starch and natural extracts. *Carbohydrate Polymers*, *176*(August), 187–194.
687 https://doi.org/10.1016/j.carbpol.2017.08.079
688 Otoni, C. G., Lodi, B. D., Lorevice, M. V., Leitão, R. C., Ferreira, M. D., Moura, M. R. d., & Mattoso, L. H. C.
689 (2018). Optimized and scaled-up production of cellulose-reinforced biodegradable composite films
690 made up of carrot processing waste. *Industrial Crops and Products*.
691 https://doi.org/10.1016/j.indcrop.2018.05.003
692 Piermaria, J., Bosch, A., Pinotti, A., Yantorno, O., Garcia, M. A., & Abraham, A. G. (2011). Kefiran films
693 plasticized with sugars and polyols: water vapor barrier and mechanical properties in relation to their
694 microstructure analyzed by ATR/FT-IR spectroscopy. *Food Hydrocolloids*, *25*(5), 1261–1269.
695 https://doi.org/10.1016/j.foodhyd.2010.11.024
696 Pradal, D., Vauchel, P., Decossin, S., Dhulster, P., & Dimitrov, K. (2016). Kinetics of ultrasound-assisted
697 extraction of antioxidant polyphenols from food by-products: Extraction and energy consumption
698 optimization. *Ultrasonics Sonochemistry*. https://doi.org/10.1016/j.ultsonch.2016.03.001
699 Rejeb, I. Ben, Dhen, N., Gargouri, M., & Boulila, A. (2020). Chemical Composition, Antioxidant Potential and
700 Enzymes Inhibitory Properties of Globe Artichoke By-Products. *Chemistry and Biodiversity*.
701 https://doi.org/10.1002/cbdv.202000073
702 Rinaldi, M., Littardi, P., Cavazza, A., Santi, S., Grimaldi, M., Rodolfi, M., … Chiavaro, E. (2020). Effect of
703 different atmospheric and subatmospheric cooking techniques on qualitative properties and
704 microstructure of artichoke heads. *Food Research International*.
705 https://doi.org/10.1016/j.foodres.2020.109679
706 Sánchez-Rabaneda, F., Jáuregui, O., Lamuela-Raventós, R. M., Bastida, J., Viladomat, F., & Codina, C. (2003).
707 Identification of phenolic compounds in artichoke waste by high-performance liquid chromatography-

tandem mass spectrometry. *Journal of Chromatography A*. https://doi.org/10.1016/S0021-9673(03)00964-6

Scarlet, D. (2013). Annex: A new Circular Economy Action Plan For a cleaner and more competitive Europe. *Journal of Chemical Information and Modeling*, *53*(9), 1689–1699. https://doi.org/10.1017/CBO9781107415324.004

Seligra, P. G., Medina Jaramillo, C., Famá, L., & Goyanes, S. (2016). Biodegradable and non-retrogradable eco-films based on starch-glycerol with citric acid as crosslinking agent. *Carbohydrate Polymers*. https://doi.org/10.1016/j.carbpol.2015.11.041

Sharma, P., Gaur, V. K., Sirohi, R., Varjani, S., Hyoun Kim, S., & Wong, J. W. C. (2021). Sustainable processing of food waste for production of bio-based products for circular bioeconomy. *Bioresource Technology*, *325*(January), 124684. https://doi.org/10.1016/j.biortech.2021.124684

Sherwood, J. (2020). The significance of biomass in a circular economy. *Bioresource Technology*, *300*(January). https://doi.org/10.1016/j.biortech.2020.122755

Silva, K. C. G., & Sato, A. C. K. (2017). Biopolymer gels containing fructooligosaccharides. *Food Research International*. https://doi.org/10.1016/j.foodres.2017.08.042

Straccia, M. C., Romano, I., Oliva, A., Santagata, G., & Laurienzo, P. (2014). Crosslinker effects on functional properties of alginate/N-succinylchitosan based hydrogels. *Carbohydrate Polymers*, *108*(1), 321–330. https://doi.org/10.1016/j.carbpol.2014.02.054

Ubeda, S., Aznar, M., Rosenmai, A. K., Vinggaard, A. M., & Nerín, C. (2020). Migration studies and toxicity evaluation of cyclic polyesters oligomers from food packaging adhesives. *Food Chemistry*. https://doi.org/10.1016/j.foodchem.2019.125918

Wang, J., Sun, B., Cao, Y., Tian, Y., & Li, X. (2008). Optimisation of ultrasound-assisted extraction of phenolic compounds from wheat bran. *Food Chemistry*. https://doi.org/10.1016/j.foodchem.2007.06.062

Wang, K., Lim, P. N., Tong, S. Y., & Thian, E. S. (2019). Development of grapefruit seed extract-loaded poly(ε-caprolactone)/chitosan films for antimicrobial food packaging. *Food Packaging and Shelf Life*, *22*(August), 100396. https://doi.org/10.1016/j.fpsl.2019.100396

734 Wrona, M., Silva, F., Salafranca, J., Nerín, C., Alfonso, M. J., & Caballero, M. Á. (2021). Design of new natural
735 antioxidant active packaging: Screening flowsheet from pure essential oils and vegetable oils to ex
736 vivo testing in meat samples. *Food Control*. https://doi.org/10.1016/j.foodcont.2020.107536
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Figure captions

Fig. 1. A- Chromatographic profile of A)- standard mixture (1-Glucose; 2-Fructose; 3-Sucrose; 4-Melezitose; 5-1-kestose; 6-nystose; 7-fructofuranosyl-nystose); B)- Chromatographic profile of a red onion byproducts sample.

Fig. 2. Total polyphenolic content expressed as gallic acid equivalents (mg/ g of dry weight) evaluated in the byproducts extracts tested. Letters indicate significant difference in ANOVA.

Fig. 3. Induction period (hours), measured by Oxitest, of A-(upper graph) sunflower oil; B-(lower graph) sunflower oil enriched with artichoke byproducts extract.

Fig. 4. Active and edible films obtained: blank film (A), artichoke (B), red onion (C) and thistle (D) based films. Lower section (E): example of an edible film applied on a beef hamburger surface.

Table 1. Amounts of carbohydrates and phenolic compounds recorded (g/100g) in vegetable byproducts*

Carbohydrates	**Onion**	**Artichoke**	**Thistle**
Glucose	27.0±1.5	2.90 ± 0.05	0.74 ± 0.04
Fructose	17.3±0.3	2.52 ± 0.04	0.59 ± 0.07
Sucrose	17.4±1.3	nd	0.72 ± 0.06
1-kestose	13.2±0.7	nd	nd
Nystose	2.7±0.1	nd	nd
Fructosylnystose	1.5±0.1	nd	nd
Phenolic compounds			
Chlorogenic acid	nd	4.30 ± 0.22	4.04 ± 0.08
Quercitrin	0.64 ± 0.03	0.74 ± 0.07	0.30 ± 0.01
Quercetin	0.27 ± 0.02	0.40 ± 0.01	0.17 ± 0.02

*Mean composition of three replicates ± SD (standard deviation).

Table 2. Induction periods (minutes) recorded by Oxitest for the different byproducts examined

Sample	Oxidation induction period (minutes)*
Sunflower oil	500 ± 13 [d]
Thistle Stems	616 ± 22 [c]
Artichoke Stems	731 ± 16 [b]
Thistle Leaves	735 ± 5 [b]
Onion Stems	1009 ± 14 [a]
Onion Leaves	1013 ± 8 [a]
Artichoke Leaves	1037 ± 49 [a]

*Mean of two replicates ± SD (standard deviation).
Superscript letters indicate comparison groups in ANOVA ($p<0.05$)

Table 3. Data from mechanical test of tensile strength of different films.

Material	MPa *
Blank film	83.2±1.2 [a]
Film thistle	87.3± 1.4 [b]
Film artichoke	96.5± 0.8 [c]
Film onion	100.9± 0.7 [d]

*Mean of two replicates ± SD (standard deviation).
Superscript letters indicate comparison groups in ANOVA ($p<0.05$)

Supplementary materials

Fig. 1S. Chromatographic profiles recorded for (A) aqueous extracts of thistle; (B) mixture of standard dextrans.

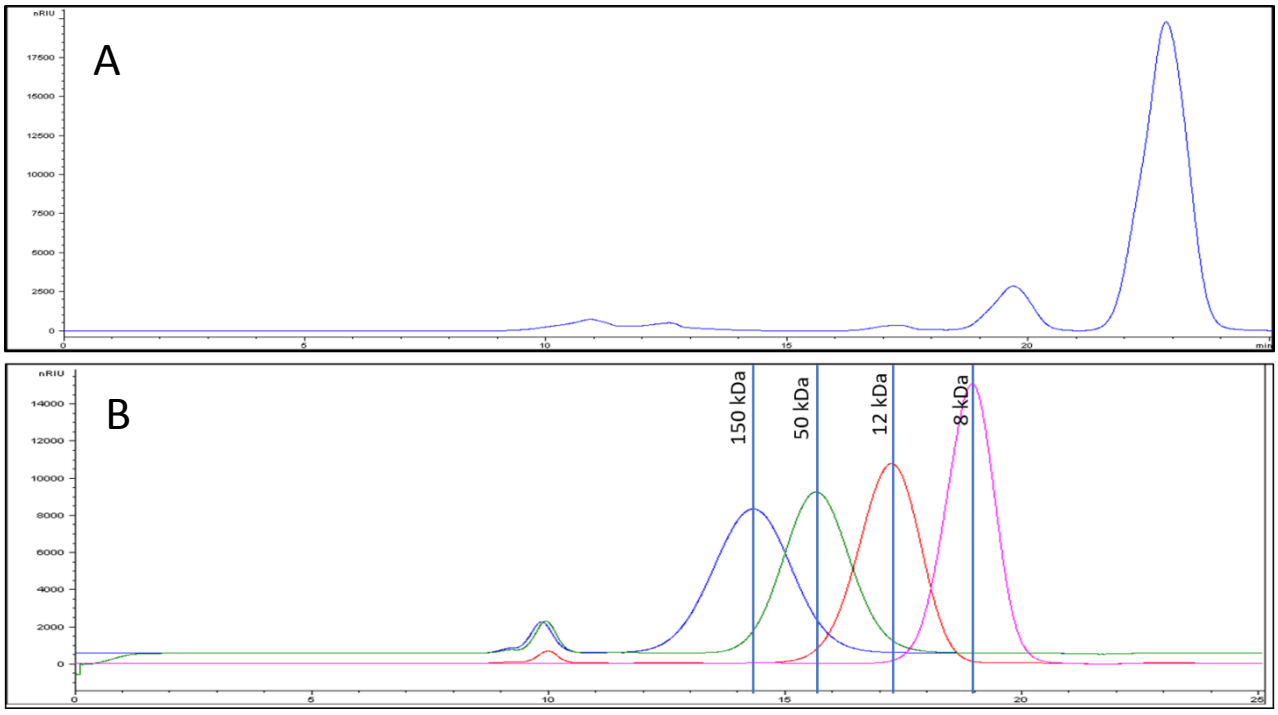

Fig. 2S. Curve showing correlation between polyphenols amount and IP values

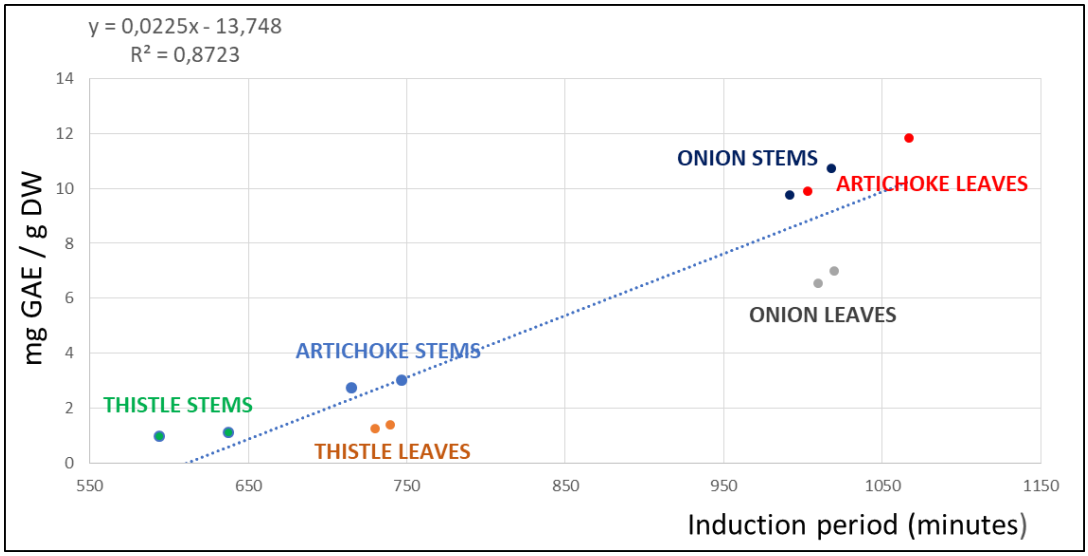

Fig. 3S. Pictures of meat samples: (A-T0) control at time 0; (A-T48h) control after 48 hours of storage at refrigerating conditions; (B-T0) sample treated with active packaging at time 0; (B-T48h) sample treated with active packaging after 48 hours of storage at refrigerating conditions

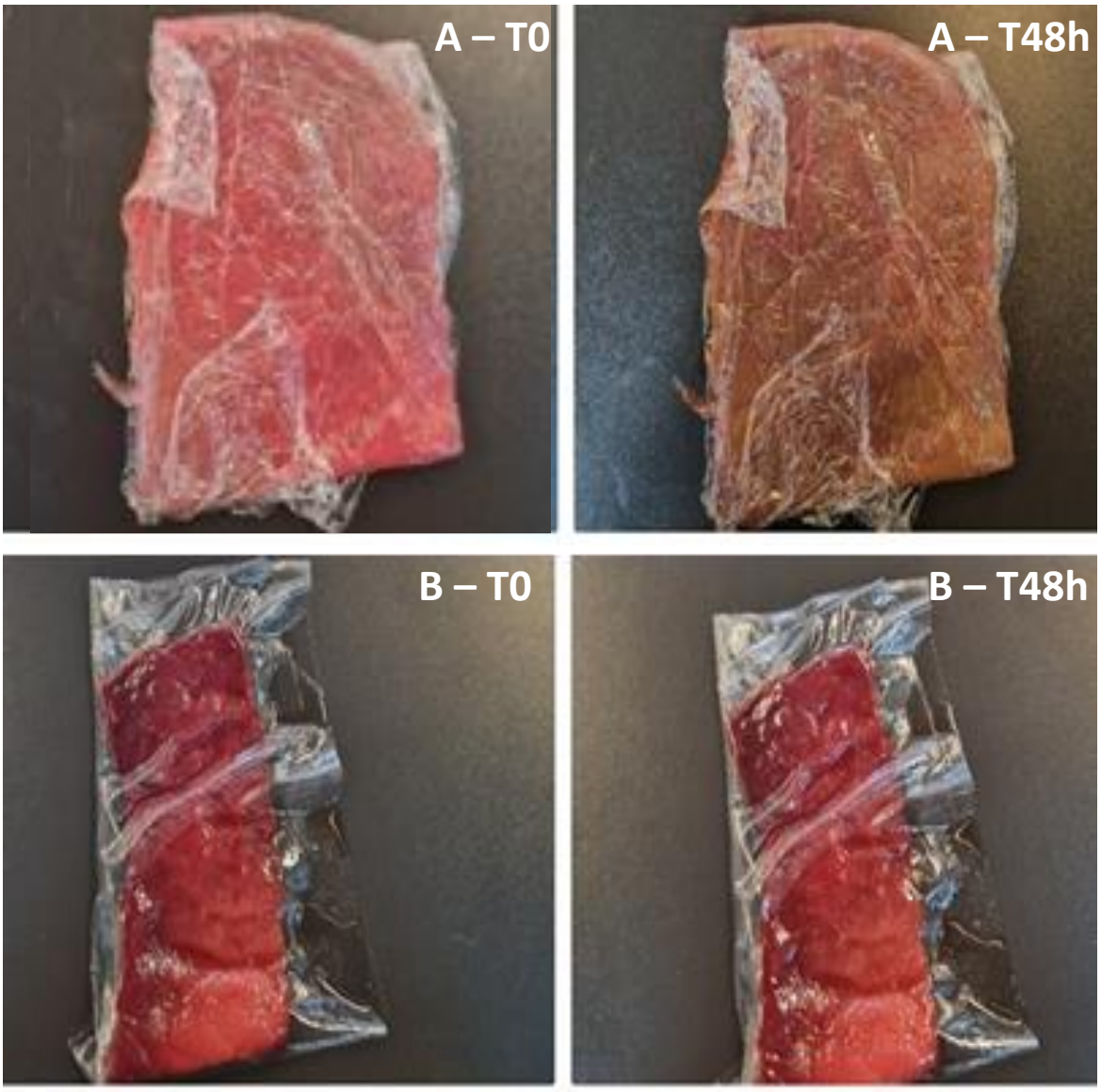

Fig. 4S. Picture of (A-T0) control fruits at T0; (A-72h) control after 72 hours of storage at room temperature; (B-T0) treated samples at T0; (B-72h) treated samples after 72 hours of storage at room temperature

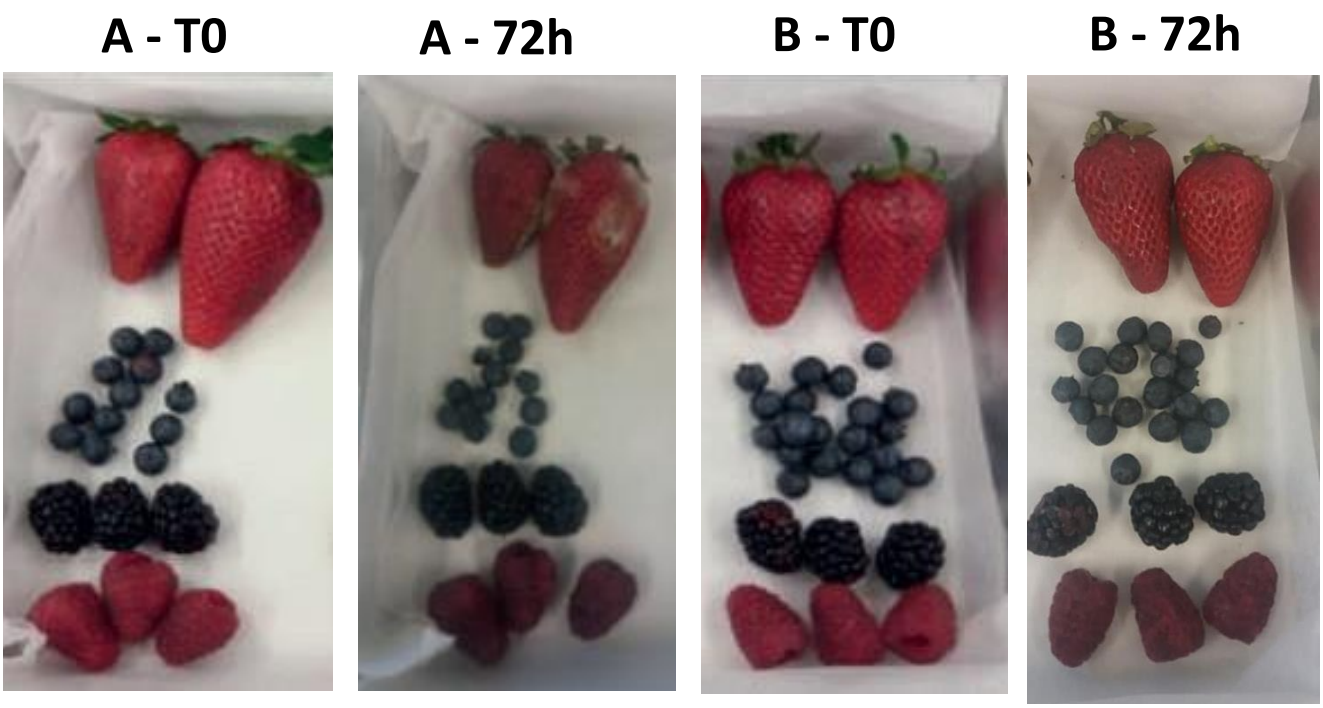

Figure 1

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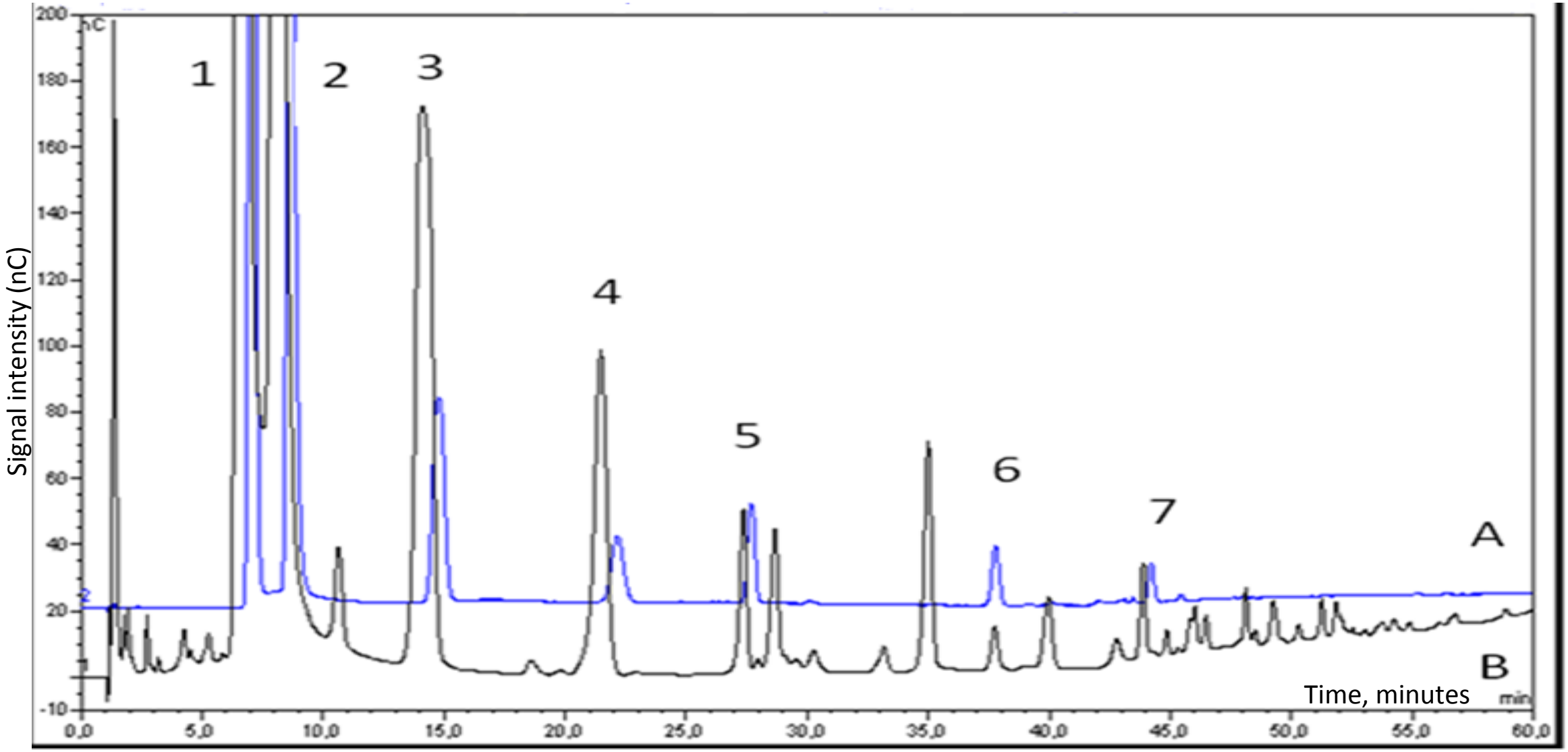

Figure 2

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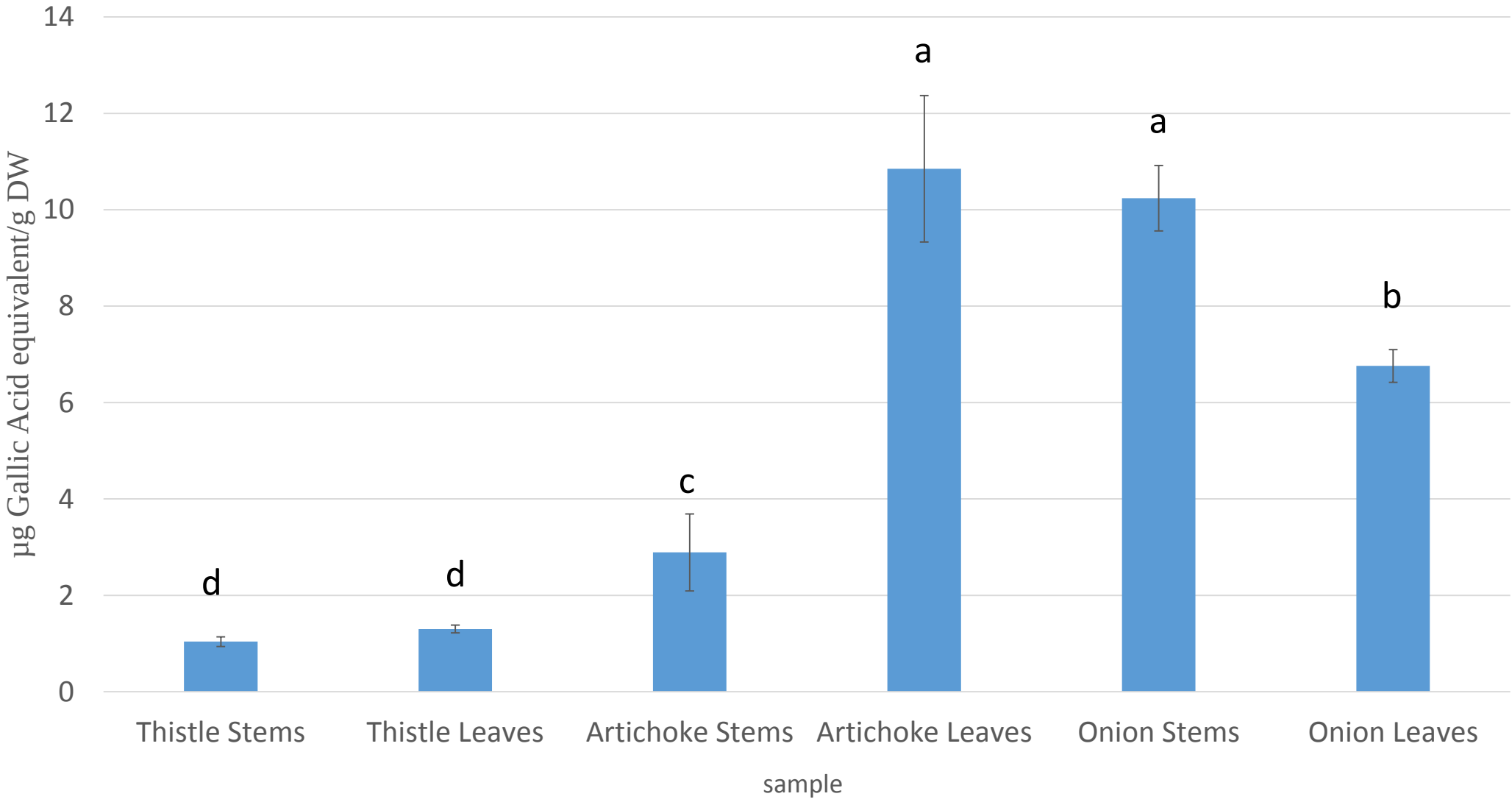

Figure 3

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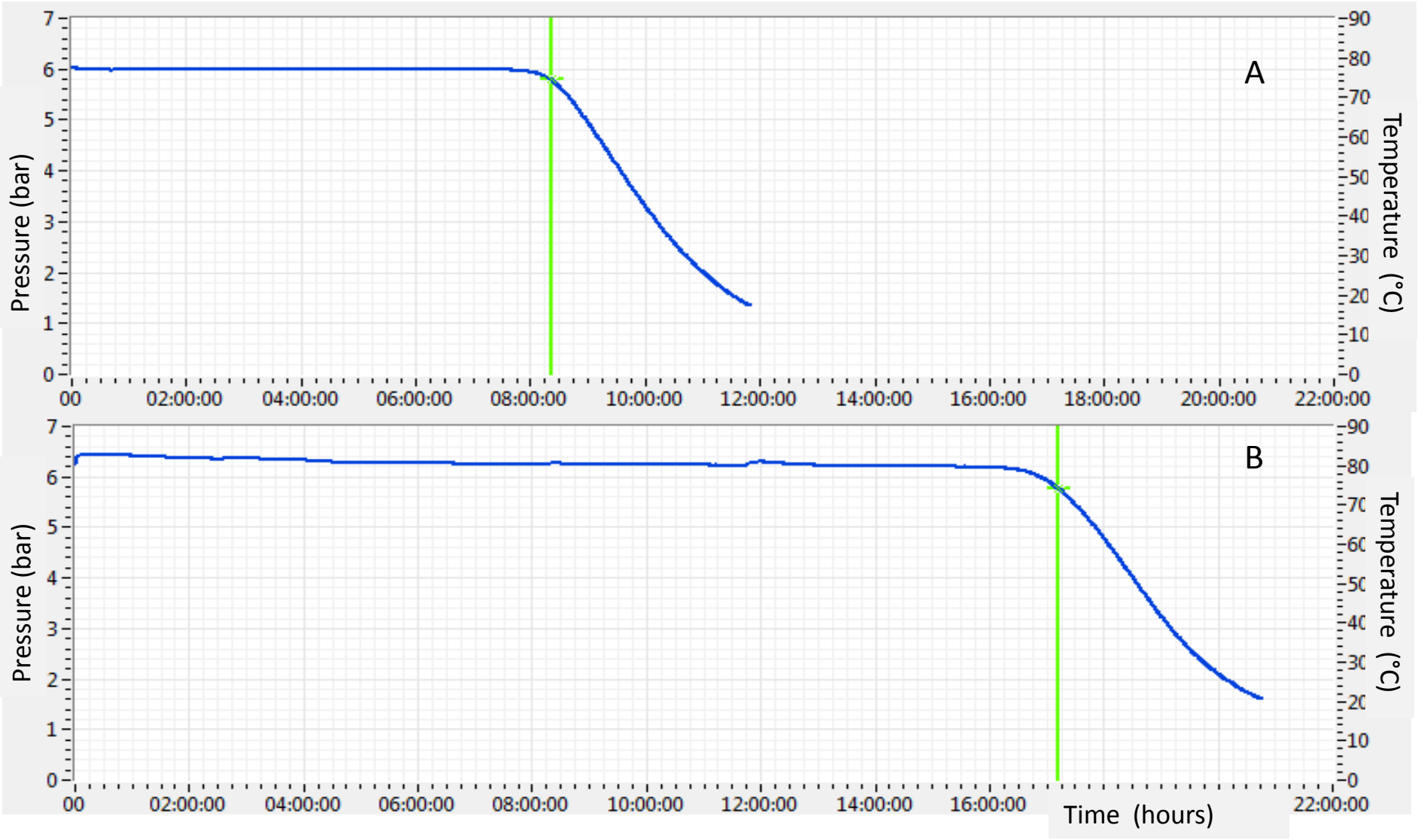

Figure 4

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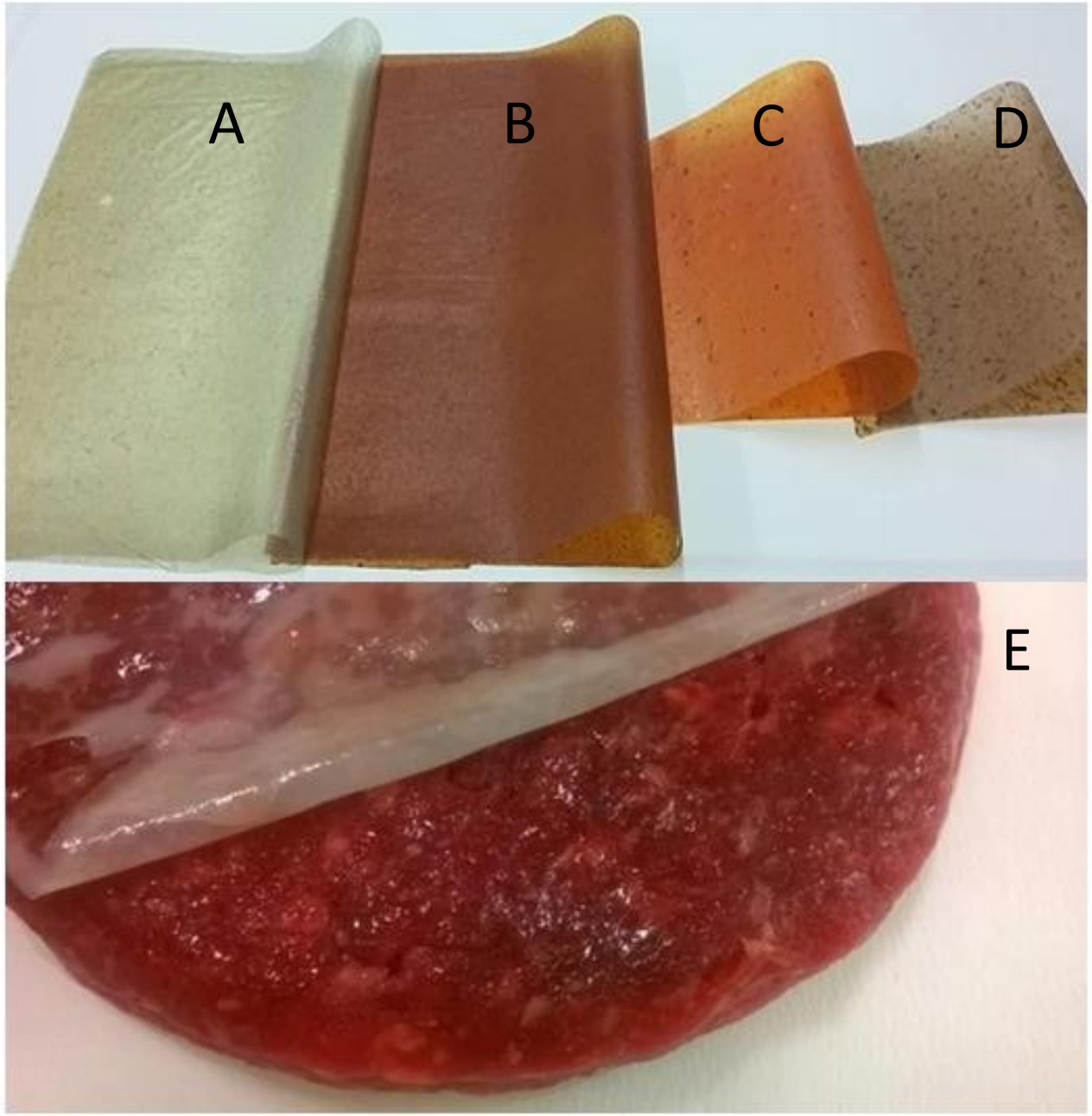

Manuscript title:

Valorization of agro-industrial byproducts: extraction and analytical characterization of valuable compounds for potential edible active packaging formulation

Authorship contribution:

Antonella Cavazza - Conception and design of the work; Data interpretation; Writing, Rewieving

Maria Grimaldi - Development of methodology; data analysis

Olimpia Pitirollo - Editing, Development of methodology; data collection

Paola Ornaghi - resources

Claudio Corradini - Critical revision of the article

All authors declare no conflict of interest

All authors approve the submission of this manuscript