Title of the Invention: Extraction of antioxidant-rich food ingredients from fruit waste

Abstract Title: Extraction of antioxidants from fruit waste

A method of processing fruit peel to provide an extract, the method comprising the steps of mixing the peel with water at a temperature in the range of 70 – 100 °C to give an aqueous mixture; allowing digestion of the fruit material in the aqueous mixture, and extracting antioxidant material from the mixture. The peel is preferably apple peel, and the antioxidants are polyphenols. The peel may be dried, and may be in the form of a powder or particulate before being mixed with water. The antioxidant material may be extracted using one or more of filtration and centrifugation. After extraction the aqueous juice may be purified by liquid-liquid partitioning, using a solvent such as ethyl acetate, or chromatographic techniques. The extract may be dried to provide a solid extract.

Fig. 1

1. Peel fruit
2. Dry Peel
3. Grind to Powder
4. Mix with Heated Water
5. Enable Digestion
6. Extraction
7. Dry Extract

INT CL:
A23L 1/212 (2006.01)  A23L 1/30 (2006.01)

Documents Cited:
US 6626452 A
US 5968517 A
US 20100331399 A1

Field of Search:
INT CL A23L
Other: Online: WPI, EPDOC
Peel fruit

Dry Peel

Grind to Powder

Mix with Heated Water

Enable Digestion

Extraction

Dry Extract

Fig. 1
EXTRACTION OF ANTIOXIDANT-RICH FOOD INGREDIENTS FROM FRUIT WASTE

Field Of the Application

The present application relates to a process for obtaining a bioactive extract from fruit and the use of this extract has use as a food ingredient.

Background Of The Application

Apples are one of the most widely produced tree fruits and the third most traded fruit, behind bananas and grapes. In 2008, 7.5 million metric tonnes of apples were exported globally, with 65% of the worldwide production being accounted for by China, the EU, Chile, and the US. Whilst the apple fruit may be consumed fresh, it is also processed by manufacturers, for example, into juices, cider or canned sauces. The waste generated during processing, i.e. mainly pomace and peels, is generally recycled as a fertiliser via land spreading or used as feedstock. As an example of the quantities involved, the US National Agriculture Statistics Service reported that 267 million pounds of apples were processed in to applesauce and canned apple in the New York State in 2001, generating an estimated 20 million pounds of peels. In Ireland, it has been estimated that in 2007 over 300 tonnes of waste Apple peels were produced and in the UK the figures has been estimated at over 5,000 tonnes.

The present application is directed at the provision of an alternative use for this waste and more specifically the peel.

It is known to recover phenolic compounds from Apples generally. The most known process involves the pressing and/or extraction of the fruit samples with liquid organic solvents (i.e. aqueous solvents such as ethanol, methanol or acetone, or combinations of them at different ratios) in order to obtain a crude juice or extract. The latter is further purified using different techniques, such as the use of solvents of different polarity or a solid phase extraction using Sep-Pak cartridges conditioned with different solvents, or other column chromatography


15 The use of organic solvents and/or aqueous mixtures thereof for the extraction of phenolics from plant sources generally leads to crude extracts with various co-extracted compounds (i.e. lipoidal material such as waxes, fatty acids or terpenes; pigments such as chlorophylls and carotenoids; sugars; organic acids and peptides).

20 The crude extracts may be further processed by a liquid-liquid partitioning systems with solvents of different polarity or solid-phase extraction systems (i.e. based on adsorption). These further processes may also be used together for the clean-up and fractionation of target phenolic compounds present in the crude mixtures. Organic solvents such as hexane, diethyl ether, chloroform, acetonitrile, petroleum ether, acetone, methanol or ethanol are generally used for such a purpose.

25 Unfortunately, the process results with residues of organic solvents. These organic solvents may be difficult to dispose of. This may be for a number of reasons including its flammable nature, toxicity for the environment and associated health hazards. Moreover, if the solvents are present in the concentrated final
purified phenolic extract, the use of the extract could be extremely restricted for human consumption.

Ethanol is among the few organic solvents with recognised dietary use, and is by far the most accepted for the extraction of phytochemical compounds (i.e. bioactive compounds from plants) for further food use. However, ethanol could represent a problem for those food applications in which the presence of alcohol as a substrate could change the characteristics of the food (i.e. changes in the indigenous microflora population or in the product category) (Shi et al., 2005). Accordingly, the present application seeks to provide an alternative process to provide a phenolic extract from fruit peel.

Summary

The present application reduces the need for organic solvents in the initial liquid extraction processing and further clean-up and instead relies upon using heated water as a means of reducing the bioburden of the plant material, while at the same time rendering the cell walls more permeable for the release of phenolic compounds that are water-soluble. As lipoidal material (i.e. waxes and terpenes) and pigments such as chlorophylls and carotenoids are poorly or not soluble in water, the resulting crude extracts do not need further clean-up with organic solvents (i.e. petroleum ether, ether, hexane and the like).

Accordingly, a first embodiment of the application provides a method as detailed in claim 1. Advantageous embodiments are provided in the dependent claims.

Brief Description Of The Drawings

The present application will now be described with reference to the accompanying drawings in which:

Figure 1 is a process flow diagram illustrating the process of production;
Detailed Description Of The Drawings

As described above, the present application provides a process in which fruit-waste, i.e. fruit peel is processed to provide an extract. The present application will now be described with reference to apple peel but may also be applied to similar fruit, such as pears.

The process of the present application is suitable for apples. Whilst it may be used with any apples, in practise the cooking or culinary apple (*Malus domestica* Borkh., Fam. *Rosaceae*) which refers to a variety that is generally eaten after being cooked is more suitable since in practise it is generally peeled before being processed into sauces, compotes or purees. One particular variety of apple that is suitable, is the cultivar Bramley's Seedling which is generally regarded as the most important cooking apple in the British Isles (Great Britain and Ireland). The Bramley apple is almost exclusively a British variety; however it is also grown in the United States; and can be found in Canada. Advantageously, the Bramley apple contains a large amount of polyphenol components of interest. It will be appreciated that it may readily be determined by experiment using, for example, the process described herein as to which other varieties of apples\fruits may be used successfully.

For the preparation of the polyphenol extract, the process begins with the peel as of the raw fruit as illustrated in the process of Figure 1. The peels from the fruit are desirably obtained in a peeling process for example by a mechanical peeling system with blades which would be familiar to those skilled in the art. It will be appreciated that other (non mechanical) physical-chemical peeling processes (i.e. by steam or by using lye solutions) are less desirable in order to avoid loss and changes in the physical-chemical composition and solubility of bioactive compounds due to heat sensitivity, oxidation, hydrolysis, and leaching effects.

The peel may then be subjected to a washing process to remove all impurities which cannot be eliminated at the processing step.

As the apple peel is perishable, it is appropriate to dry the peel to preserve it as there may be some time between the initial peeling process and the subsequent steps and some phenolics are unstable or degrade by enzyme action
in undried plant material. The drying step 4 may be a thermal or non-thermal drying process and examples would include oven-drying, freeze-drying, air-drying or drum-drying.

Once dried, the bulk peel material may be reduced into smaller particles using a suitable process, for example by crushing, grinding or shredding. This reduction in the particle size of the peel increases the efficiency of the subsequent extraction process. As an example, a mechanical grinding system may be employed to powder the dried bulk peel material.

Commencing with the dried powdered apple peel, an exemplary process of the present application provides the steps outlined below to process the peel to provide an extract. It will be appreciated that not all of these steps are essential as explained further below. In brief, the extraction process comprises the following steps:

1. Water extraction (8) of peel powders;
2. Plant digestion (10) at constant temperature (suitably less than 100°C);
3. Process to provide crude peel juices;
4. Isolation of phenolic compounds (purified extract);
5. Freeze-drying; and
6. Antioxidant ingredient;

In the first steps (1-3), the peel powder is extracted in hot water to provide an aqueous crude juice.

For the extraction step (1), the water is added to the peel in a suitable container. Suitably, the temperature of the water is in the range 80°C-100°C (maximum limits). More desirably, the range is between 85°C-95°C. Most suitably, the temperature of the water is 90°C. The use of pre-heated water as opposed to adding to cooler water and heating helps the enzymatic inactivation (polyphenol oxidase and the like) that otherwise affects the polyphenol substrates (i.e. browning development, and transformation into different compounds). The quicker the inactivation, the lower the browning phenomena.
For the plant digestion step (2), the temperature of the heated peel material is kept constant for a period of time between 10 and 30 minutes, more desirably about 20 minutes, by using a suitable heat transfer system, for example using conduction or convection mechanisms. Suitably, the digestion vessel is maintained under reduced air pressure (~10 mbar) throughout the heat transfer process. Desirably, the digestion vessel is kept under a gentle continuous shaking or stirring.

In steps 3 and 4, the digested peel material is subjected to one or more extraction 12 steps, for example filtration and/or centrifugation may be performed as step 3 to provide crude juices. Suitably after this step a clear aqueous juice is the end result.

In step 4, the resulting aqueous juice may be purified by liquid-liquid partitioning with a low-boiling organic solvent with low-medium polarity and food-grade characteristics (e.g. ethyl acetate) as to obtain a purified polyphenol extract.

Other suitable clean-up and fractionation systems include column chromatography techniques, i.e. solid-phase extraction based on liquid-solid adsorption of target compounds on non-ionic resins with non-polar bonded phases (e.g. octadodecyl, C₁₈).

For this purification step with partitioning with organic solvents, ethyl acetate is preferred over similar low-polar or non-polar solvents (i.e. dimethylsulphoxide, dichloromethane, diethyl ether) because of its accepted use in dietary applications. The solubility of the compounds and their distribution within the aqueous (juice) and the organic solvent phases can be controlled in relation to their acidity characteristics, by modifying their degree of protonation with regard to the pH of the juice. For this step, the aqueous juice is neutralised (pH 7) with basic solutions, desirably using a strong mineral base (sodium hydroxide) as a means of increasing the yield of recovery. The purified polyphenol extract may be further concentrated by evaporating the organic solvent under reduced pressure until dry. The organic solvent may be distilled and recycled for further extractions.

The purified polyphenol extract may be recovered as a liquid preparation by solubilising the solids in water, ethanol, or mixtures thereof. The liquid
preparation may be further processed, i.e. either as it is or after an auxiliary agent for powdering such as dextrin or the like is added, into a powder preparation by drum-drying, spray-drying or freeze-drying.

The crude juice extracted after digestion contains organic acids and sugars and is slightly acidic with a pH around 4.5. Due to the presence of such nutrients and to the fact that the juice has been mildly pasteurised (90°C, for 20 minutes) but not sterilised, it may still be subject to spoilage microorganisms, such as yeasts and fungi.

Accordingly, a further process 14 may be included to prolong shelf life. One such process would be employ drying to prevent such spoilage mechanisms although other processes may be used as would be familiar to those skilled in the art. From a nutritional point of view, the juice from the peels contains mainly organic acids, such as vitamin C, and phenolics. The heat process employed in thermal drying can destroy most vitamins, but it does not generally affect the phenolic compounds.

The fruit waste-derived polyphenol obtained in the present invention comprises of monomeric flavan-3-ols (catechins), flavonols (quercetin glycosides), dihydrochalcones (phloretin glycosides) and the like as simple polyphenol compounds and oligomeric flavan-3-ols (procyanidins) and the like as high-molecular polyphenol compounds. Phenolic acids typically found in the raw fruit material, i.e. hydroxycinnamic acids such as caffeic acid and p-coumaric acid derivatives, are generally not present or scarcely represented in the purified polyphenol extract thus obtained. Their absence can be seen as advantageous as phenolic acids such as chlorogenic acid are ideal substrates for enzymatic browning in fruits and/or vegetables, and related processed products (i.e. juices or beverages).

This fruit polyphenol extract can be added to juices, beverages and foods in general so that they can be suitably used as natural ingredients with antioxidant activity and preservative capacity, in partial or total substitution of artificial food
additives for which a dose-dependent hazardous effect is documented (i.e. BHA, BHT, TBHQ) and/or the addition level is critical for the quality and final acceptance by the end consumer (i.e. sulphite preservatives causes severe allergic reactions in sensitive people).

The present application allows for the preparation of a purified fruit polyphenol ingredient with no characteristic odour related to the raw fruit material, as the volatile fraction is evaporated together with the organic solvent when the fruit polyphenol extract is concentrated. The ingredient can be directly solubilised in liquid foods such as fruit or vegetable beverages and juices; it can be also applied as a powder to dry foods such as breads, snacks or candies.

The process generates an odourless powdered substance with applications in the food ingredient market as a natural antioxidant or preservative.

Its effectiveness is on par with the main commercial preservatives and antioxidants used in the food industry. This technology has a number of commercial advantages:

- A safer more natural alternative to synthetic additives (e.g. THB, sulphites, TBHQ etc)
- Extended food applications, as existing phenolic compound extractions leave solvent residue
- A more environmentally friendly sustainable process
- Comparable yield and cost
- Industrial applications for phenolic antioxidants are varied (i.e. food, healthcare, pharmaceutical, cosmetics, etc) because oxidation is critical for the preservation of vitamins, fats, oils and emulsions

The present application provides a number of advantages including for example the use of an abundant solvent (water) with traditional and safe use for human consumption as for the solvent extraction. Moreover, there is limited co-extraction of undesirable lipoidal materials, such as waxes, terpenes and plant pigments in the crude aqueous juice. The limited co-extraction (practically none)
of the plant pigments, e.g. chlorophylls and carotenoids, avoids the requirement for subsequent depigmentation steps, which generally involve using non-environmentally friendly and/or high flammable organic solvents such as hexane, chloroform or petroleum ether.

The output of the process is an antioxidant-rich ingredient with applications in the food and drink industry, as a novel food ingredient and/or preservative.

The words comprises/comprising when used in this specification are to specify the presence of stated features, integers, steps or components but does not preclude the presence or addition of one or more other features, integers, steps, components or groups thereof.
Claims

1. A method of processing fruit peel to provide an extract, the method comprising the steps of:
   mixing the fruit peel with water at a temperature in the range 70°C to 100°C to provide an aqueous mixture;
   allowing the digestion of the fruit material in the aqueous mixture;
   and extracting antioxidant material from aqueous mixture.

2. A method of processing fruit peel according to claim 1, wherein the peel is dried before mixing with the water;

3. A method of processing fruit peel according to claim 1 or claim 2, wherein the fruit peel is reduced in size.

4. A method of processing fruit peel according to claim 3, wherein the fruit peel is reduced in size to a powder or particulate.

5. A method according to claim 4, wherein the step of reducing comprises the use of a mechanical grinding system.

6. A method according to any preceding claim, wherein the step of extracting comprises the use of one or more of filtration and centrifugation on the aqueous mixture.

7. A method according to any preceding claim, wherein the step of extracting comprises drying of said liquid extract to provide a solid extract.

8. An antioxidant extract obtained from apple peel by the method of any preceding claim.
Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

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<th>Category</th>
<th>Relevant to claims</th>
<th>Identity of document and passage or figure of particular relevance</th>
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<td>1-8</td>
<td>US 6620452 A (AMERICAN FRUITS) see paragraphs [0019] - [0034] in particular</td>
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<td>US 5968517 A (DUNCAN) see column 3 line 30 - column 4 line 60 in particular</td>
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<td>US 5912363 A (NAFISI-MOVAGHAR) see column 4 lines 10-30 in particular</td>
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- & Member of the same patent family
- A Document indicating technological background and/or state of the art.
- P Document published on or after the declared priority date but before the filing date of this invention.
- E Patent document published on or after, but with priority date earlier than, the filing date of this application.

Field of Search:
Search of GB, EP, WO & US patent documents classified in the following areas of the UKC:

Worldwide search of patent documents classified in the following areas of the IPC
A23L

The following online and other databases have been used in the preparation of this search report
Online: WPI, EPODOC

International Classification:

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