Extraction, isolation and utilization of bioactive compounds from fruit juice industry waste

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CHAPTER 9 Extraction, Isolation and Utilization of Bioactive Compounds from Fruit Juice Industry Waste

9.1 Introduction

Juice manufacturing is an important segment of the food industry. Within the fruit and vegetable drink market, approximately 50%, 30%, and 20% of the market share belong to: juice drinks mixed with a pure juice (0-24% juice content), pure juice (100%), and nectars (25-99% juice content), respectively. In 2011, the global consumption volume of commercial juices and nectars are approximately 39 billion litres, which was equivalent to approximately USD 107 billion in market value (AIJN 2014). The most popular juices are orange and apple; others include juices from lemon, grape, grapefruit, peach, pomegranate, berries, and exotic fruits, such as pineapple, mango, mangosteen, and passion fruit (McLellan and Padilla-Zakour 2004, AIJN 2014, Reyes-De-Corcuera et al. 2014). Nevertheless, with the success and high growth of the functional food products in the last decade, demands for juice products with health benefits continues to rise, as is the demand for products in a variety of packages with increased emphasis on functionality, new flavours or blends. With the drives of new production and packaging technology, together with the launch of new super-premium juices, the global market of the juice industry is still expecting a steady growth (AIJN 2014, López 2014, Leatherhead Food Research 2014).

With high production volume, inevitably juice industry generates a large quantity of waste as a consequence. Waste streams from fruit juice processing are produced both in solid and liquid forms (McLellan and Padilla-Zakour 2004). Liquid waste streams are mainly discharge of cleaning water and process water which has low-to-medium biological oxygen demand (BOD) values and can be treated by aerobic or anaerobic systems (Arvanitoyannis and Varzakas 2008). Solid waste, on the other hand, is highly polluted and more difficult to treat (Kosseva 2011). Conventionally these wastes are disposed by means of using as animal feeds or fertilizers (Van Dyk et al. 2013). Although they are discarded from the process as they cannot be further utilized, fruit solid wastes retain high concentrations of
several bioactive compounds. The peels of several fruits (for example apple, peach, pomegranate) contain higher amount of bioactive compounds than the edible parts (Gorinstein et al. 2001, Li, Guo et al. 2006). Substantial evidence points out that all parts of fruit solid wastes are rich in health-benefit phytochemicals (Widmer and Montanari 1994, Balasundram et al. 2006, Ayala-Zavala et al. 2011, O'Shea et al. 2012, Dhillon et al. 2013, Mirabella et al. 2014). Rather than using them conventionally for feeds and fertilizers, alternative valorisation of these unwanted materials to create higher value-added products is a better option, and this topic has attracted great interest among researchers and industry alike in the last few decades.

This chapter will focus on the recovery of bioactive compounds, particularly phenolic compounds and dietary fibre, from fruit juice industry solid wastes. It aims to provide a comprehensive information on the use of such wastes as a source of high value-added components. Extraction, isolation and potential applications of phenolic compounds and dietary fibre recovered from fruit solid wastes in the food industry will be discussed.

9.2 Waste from fruit juice industry

The types of fruits for juice processing can be broadly classified into pome fruit (e.g. apple, pear), citrus (e.g. orange, lemon, lime, tangerine, grapefruit), stone fruits (e.g. peach, nectarines, cherry), berries (e.g. grapes, pomegranate, cranberry, blackcurrant), and exotic fruit (mango, pineapple, mangosteen, passion fruit). Manufacturing of fruit juices consists of a series of unit operations which varies depending on the nature of raw materials and the characteristics of the desired final products (McLellan and Padilla-Zakour 2004). A general process includes pre-treatment steps, juice extraction, and post-extraction treatment steps. Figure 1 illustrates a general flow diagram of juice processing process according to fruit type including operation steps where solid wastes are generated. Detailed manufacturing, including the objectives of each step, of juices from different types of fruit can be found in the literature (McLellan and Padilla-Zakour 2004, Horváth-Kerkai and Stéger-Máté 2013, Reyes-De-Corcuera et al. 2014).
Figure 1 General flow diagram of juice manufacturing according to fruit types. Thick solid grey lines represent common unit operations in all fruit juice processing. Dash and solid thin black lines represent variations in processing processes between different fruit types. Unit operations with a star on the top right indicate the point where solid wastes are generated.
Solid waste from fruit juice manufacturing is generated throughout the processing line. They are parts of raw materials that cannot be utilised in the production of the intended products (Commission Regulations 442/1975/EEC; 689/1991/EEC), which include pomace, peels, seeds, stones, stems. Estimation of manufacturing fruit waste is not straightforward since there is no distinct universal definition of food waste (Monier 2011, Buzby and Hyman 2012). According to a report published by the Food and Agriculture Organization, the 2007 production volume of fruits and vegetables worldwide was 1,650 million tonnes, of which approximately 12% (or 198 million tonnes) was wasted at processing stage (Gustavsson et al. 2011). Geographically, high percentages of fruit and vegetable manufacturing wastes (20-25%) were generated in Sub-Saharan Africa, North Africa, West and Central Asia, South and Southeast Asia, and Latin America, while those percentages in Europe, North America and Oceania, and Industrialized Asia were small (2%) (Gustavsson et al. 2011). Raw materials important to juice industry at a global scale include citrus (orange), pome fruit (apple), stone fruits, berries, grape, and exotic fruits (pineapple and apple). The nature and approximate percentages of these wastes from juice manufacturing are shown in Table 1.

Of all the fruits important to the international juice trade, citrus, particularly oranges, is the largest fruit crop and juice produced worldwide. Apart from oranges, other fruits of importance include lemons, limes, grapefruits, tangerine and mandarins. With only approximately 50% juice recovery from fresh weight, a considerably high quantity of citrus pomace (50% peel composed of albedo and flavedo, 0.1-5% seeds, pulp, carpellary membrane) are generated as waste (Rezzadori et al. 2012). On dry weight basis, citrus pomace contains high contents of sugars, protein, essential oil (peel and seeds), pectin (highest concentration in peel), and dietary fibres (Marín et al. 2007).

In second place after citrus, apple juice industry also generated several million tonnes of solid waste (Bhushan et al. 2008). Apple pomace accounts for 25-30% of the total processed fruits weight, and consists of peel, core, seed, calyx, stem and soft tissue (Foo and Lu 1999). Fresh pomace contains high moisture content (70-80%) and is highly perishable as high amount of carbohydrates (10-22%, with up to 50% fermentable sugars) is present (Gullón et al. 2007, Dhillon et al. 2013).
TABLE 1 Global production quantities of fruits and juices in 2013, and approximate percentages and nature of solid waste generated from fruit juice manufacturing

<table>
<thead>
<tr>
<th>Fruit</th>
<th>Total quantity of fruit produced (tonnes)</th>
<th>Total quantity of juice produced1 (tonnes)</th>
<th>Solid waste</th>
<th>Approximate percentage of waste from raw material (w/w)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Apples</strong></td>
<td>80,822,521</td>
<td>569,962 1,452,370 (c)</td>
<td>Pomace, skin, seeds, stem</td>
<td>25-30</td>
</tr>
<tr>
<td><strong>Citrus</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Total</td>
<td>135,169,941</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Oranges</td>
<td>71,305,973</td>
<td>2,133,190 1,697,084 (c)</td>
<td>Pomace, peel, seeds</td>
<td>50</td>
</tr>
<tr>
<td>- Lemons and limes</td>
<td>14,949,082</td>
<td>96,913 (lemon) 83,740 (lemon, c)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Grapefruits</td>
<td>8,255,486</td>
<td>233,177 115,157 (c)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Tangerines and mandarins</td>
<td>28,666,714</td>
<td>2,381</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Grapes</strong></td>
<td>77,181,122</td>
<td>761,712</td>
<td>Pomace, skin, seed, stems</td>
<td>20</td>
</tr>
<tr>
<td><strong>Berries</strong></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>- Cranberries</td>
<td>540,259</td>
<td>N/A</td>
<td>Pomace, skin, seed, stem</td>
<td>5</td>
</tr>
<tr>
<td>- Currants</td>
<td>706,910</td>
<td>N/A</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Stone fruits</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Peaches and nectarines</td>
<td>21,638,953</td>
<td>N/A</td>
<td>Pomace, skin, stones, stems</td>
<td>N/A</td>
</tr>
<tr>
<td>- Cherries (sweet and sour)</td>
<td>3,643,083</td>
<td>N/A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Plums</td>
<td>11,528,337</td>
<td>6 (c)</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Exotic Fruits</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>- Pineapples</td>
<td>24,778,262</td>
<td>941,177 331,575 (c)</td>
<td>Skin, core</td>
<td>33-50</td>
</tr>
<tr>
<td>- Mangoes and mangosteens</td>
<td>42,663,770</td>
<td>255,162 (mango)</td>
<td>Peel, stone</td>
<td>35-60</td>
</tr>
</tbody>
</table>

1 Numbers without (c) are quantities of single strength juices; numbers with (c) signify quantities of concentrated juices.

Grape juice is not as highly popular among consumers as orange and apple juices are (AIJN 2014, Reyes-De-Corcuera et al. 2014). Indeed, the majority (80%) of fresh grape produced goes to wine making (Martí et al. 2014). Grape juice is not normally consumed in large amounts alone because it is either too sweet (about 200 g / L sugars) or too acidic (up to 10 g / L tartaric acid) and usually blended with other juices for a more balanced taste and flavour (Kashyap et al. 2001). Nonetheless, together with wine production, several million tonnes of grape residue are produced annually (Oreopoulou and Tzia 2007). After juice pressing (both in the wine or juice manufacturing) approximately 20% of processed grape are discarded. The residue consists of 10-20% grape pomace and 3-6 % stalks (Martí et al. 2014).

Berry juices are marketed as ‘Superfruit’ juices and interest in consumption of food in this category has increased (López 2014). Different berries (blueberry, raspberry, strawberry, currants, and pomegranate) are processed as juices. Generally, in berry juice manufacturing, solid wastes usually come from pre-treatment (washing and sorting) and juice pressing. The wastes from pre-treatment stage consist of damaged fruits, stems and stalks, while that from pressing is pomace (Tomás Barberán 2007). Percentages of berry wastes vary, depending on the nature of the fruits. For example, cranberry solid waste (pomace, stems) is 5% of processed fruit weight (Arvanitoyannis and Varzakas 2008); while pomegranate solid waste (husk, membrane, seeds) is 50% of processed fruit weight (Tomás Barberán 2007).

Although the word production volumes of stone fruit juices are not large in global scale (FAOSTAT 2016), plum, peach, apricot and cherry, are well used and popular for juice production particularly in Europe (AIJN 2014). Like in berry juice manufacturing, solid wastes of stone fruits are generated during pre-treatment and juice pressing steps. The wastes include damaged fruits, stems, stalks, and pomace.

Exotic fruit juice manufacturing is another segment that generates a considerable quantity of waste. Pineapple, mango, and passion fruit are among the most important fruits for juice industry (Schieber et al. 2001, Mirabella et al. 2014). Exotic fruits popular for juice manufacturing (e.g. pineapple, mango, passion fruit) have high percentages of inedible/unusable parts. Passion fruit waste
could be as high as 75% of raw material as it has thick rind which accounts for 90% of the waste (Arvanitoyannis and Varzakas 2008). Although passion fruit seeds are edible, they are not a part of the final products and are removed as waste (Chau and Huang 2004).

Typically, disposal of fruit solid waste may be achieved by incineration or utilisation as animal feeds and fertilisers (Van Dyk et al. 2013). Only in some cases fruit wastes are used as raw material to produce secondary products in industrial scale. For example, grape seeds have long been known for their oil-rich characteristics, with the first mention of grape-seed oil as a possible industry made probably in 1780 (Rabak 1921). Apart from traditional uses as feeds and fertilisers, in some developing countries those wastes may be simply discarded on the outskirts of the cities, causing major pollution to the environment, or disposed of in local landfills (McLellan and Padilla-Zakour 2004). Disposal of fruit wastes incurs a very high cost to the industry. In the USA alone, disposal fee of apple pomace has been estimated to be higher than USD 10 million annually (Shalini and Gupta 2010). With regards to their used as animal feeds, not all fruit wastes are suitable for animal feeds as they may contain too low protein or too high lignin content (Van Dyk et al. 2013). Most fruit solid wastes also contain high moisture content, which requires drying to prolong their shelf-life if they are going to be used further. Energy and transport costs together with low sales prices make return on investment unattractive and this has led to alternative valorisation concepts (Laufenberg et al. 2003).

The interest in the alternative use of waste streams to create high value products beyond disposal or fertilisation has increased drastically in the last few decades. With the high growth of functional food and waste utilization concepts, fruit solid wastes have been heavily researched as cheap sources for bioactive compounds. Substantial evidence has established that fruit solid wastes retains a wide range of high-value functional compounds. With the continuity of new discoveries on extractions, isolation, and characterization techniques, the use such wastes as cheap raw materials to produce bioactive compounds in commercial scale becomes tangible in large scale.

9.3 Bioactive compounds from fruit juice industry waste
Bioactive compounds from fruits (also known as phytochemicals) possess certain biological activities – namely, antimicrobial, anticancer, anti-inflammatory, immuno-stimulatory, and antioxidant activity – from which can exert physiological effects and may enhance the human’s health (Hollman and Katan 1999, Szajdek and Borowska 2008, González-Molina et al. 2010, Johnson 2013). There are many classes of bioactive compounds, which are categorized based on their molecular identity or biopolymer constituents (Campos-Vega and Oomah 2013). Figure 2 illustrated classification of prominent functional compounds recovered from fruit solid wastes that have been extensively investigated.

**Phenolic Compounds**

Phenolic compounds are a broad group of chemical components and are structurally diverse (Naczk and Shahidi 2004). They are secondary metabolites found in plant species and more than 8,000 phenolic compounds have been identified (Croteau et al. 2000). Major classes of phenolic compounds found in fruit wastes include flavonoids (flavonols, flavones, flavonones, flavanols, anthocyanins), phenolic acids (hydroxybenzoic acids, hydroxycinnamic acids), tannins, stilbenes and lignans (Balasundram et al. 2006, Ignat et al. 2011, Gnanavinthan 2013). Flavonoids are the largest class of phenolic compounds with over 4000 identified substances, (Ignat et al. 2011). Molecular structure of phenolic compounds is found to be an important determinant of their scavenging capacity and oxidation potential (Shi et al. 2001). Several papers on the bioavailability and metabolism of various phenolic compounds have been published (Hollman and Katan 1999, Scalbert and Williamson 2000, Felgines et al. 2003, Larrosa et al. 2009).
Figure 2 Major classes of bioactive compounds recovered from fruit juice industry wastes.

*Citrus* 
(Peel, Seed)

*Peel*
<table>
<thead>
<tr>
<th>Solid fruit waste</th>
<th>Phenolic compounds</th>
<th>Dietary fibre</th>
<th>Others bioactive components</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apple pomace/</td>
<td>catechin, epicatechin, caffeic acid, chlorogenic acid, p-coumaric acid, rutin derivatives, 3-hydroxyphloridzin, phlorerin 2'-xyloglucoside, phloridzin, quercertin-glycosides</td>
<td>60-90% TDF; Cellulose, hemicellulose, lignin, pectin (12%)</td>
<td>Terpenes (ursolic acid)</td>
<td>Lu and Foo (1997), Schieber et al. (2003), Nawirska and Kwaśniewska (2005), Bai et al. (2010), Çam and Aaby (2010), Pingret et al. (2012), Reis et al. (2012), Grigoras et al. (2013), Sun-Waterhouse et al. (2013)</td>
</tr>
<tr>
<td>Apple skin</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Apple seed</td>
<td>Phloridzin, Chlorogenic acid, p-coumatylquinic acid, quercertin-glycosides, 3-hydroxyphloridzin, phlorerin 2'-xyloglucose, tocopherols ($\alpha$, $\beta$, $\gamma$, $\delta$)</td>
<td>20-24% Oil (linoleic acid, oleic acid)</td>
<td></td>
<td>Lu and Foo (1998), Schieber et al. (2003), Tian et al. (2010), Górnaś (2015)</td>
</tr>
<tr>
<td>Citrus peel</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Citrus seed</td>
<td>Eriocitrin, hesperidin, naringin, narirutin, neoerocitrin, caffeic acid, p-coumaric acid, ferulic acid, sinapinic acid,</td>
<td></td>
<td>Terpenes (limonin, nomilin, nomilin-17-\beta-d-glucoside)</td>
<td>Ozaki et al. (1991), Ohta et al. (1993), Bocco et al. (1998), Yu et al. (2005)</td>
</tr>
</tbody>
</table>
Table 2 Selected Reports on major bioactive compounds in solid wastes from fruit juice industry (continued)

<table>
<thead>
<tr>
<th>Solid fruit waste</th>
<th>Phenolic compounds</th>
<th>Dietary fibre</th>
<th>Others bioactive components</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grape pomace/</td>
<td>Anthocyanins, catechins, epicatechin, gallic acid, rutin, quercetin and kaempferol,</td>
<td>65-80% TDF; Cellulose, pectin, hemicellulose, lignin</td>
<td></td>
<td>Souquet et al. (2000), Kammerer et al. (2005), Pinelo et al. (2005), Llobera and Cañellas (2007), Ruberto et al. (2007), Rockenbach et al. (2011)</td>
</tr>
<tr>
<td>Grape skin</td>
<td>epicatechin gallate, epigallocatechin</td>
<td></td>
<td></td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>Grape seed</td>
<td>Catechin, Epicatechin, gallic acid, Epicatechin gallate, Epigallocatechin gallate,</td>
<td>Cellulose, pectin, hemicellulose, lignin</td>
<td>7-19% Oil (linoleic acid, oleic acid)</td>
<td>Molero Gómez et al. (1996), Guendez et al. (2005), Bozan et al. (2008), Köhler et al. (2008), Spranger et al. (2008), Delgado Adámez et al. (2012), Prado et al. (2012), Da Porto et al. (2013)</td>
</tr>
<tr>
<td></td>
<td>Epigallocatechin gallate, Epigallocatechin, Procyanidins , resveratrol</td>
<td></td>
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<tr>
<td>Grape stem</td>
<td>Quercetin 3-glucuronide, catechin, caffeoyltartaric acid, dihydroquercetin 3-</td>
<td>Cellulose (30.3%), hemicelluloses (21.0%), lignin (17.4%),</td>
<td></td>
<td>Souquet et al. (2000), Rayne et al. (2008), Ping et al. (2011), Prozil et al. (2012)</td>
</tr>
<tr>
<td></td>
<td>rhamnoside (astilbin), tannins, resveratrol, viniferin</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pomegranate peel/</td>
<td>anthocyanins, ellagitannins (ellagic acid, gallic acid and punicalagin), gallo-</td>
<td>30-60% TDF (cellulose, Klason lignin, uronic acid, pectin)</td>
<td></td>
<td>Cerdá et al. (2003), Lansky and Newman (2007), Fischer et al. (2011), Johanningsmeier and Harris (2011), Fawole et al. (2012), Ismail et al. (2012), Hasnaoui et al. (2014)</td>
</tr>
<tr>
<td>Pomegranate mescarp</td>
<td>tannins, gallagyl esters, hydroxybenzoic acids, hydroxycinnamic acids and dihyd-</td>
<td></td>
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<td></td>
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<tr>
<td></td>
<td>roflavonol,</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pomegranate seed</td>
<td>Gallic acid, Ellagic acid γ-tocopherol</td>
<td>Lignins, lignin derivatives                                                   Sterols (daucosterol, campesterol, stigmasterol, β-sitosterol)</td>
<td>(Dalimov et al. (2003), Wang et al. (2004), Lansky and Newman (2007)</td>
<td></td>
</tr>
<tr>
<td>Solid fruit waste</td>
<td>Phenolic compounds</td>
<td>Dietary fibre</td>
<td>Others bioactive components</td>
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<td>----------------------</td>
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<td>---------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Mango peel</td>
<td>Anthocyanins, quercetin-glycosides, kaempferol-glycoside, xanthone-glycosides, cyanidin 3-O-galactoside anthocyanidin hexoside, γ-tocopherol, Quercertin, mangiferin pentodise Syringic, ellagic, gallic Condensed tannins</td>
<td>30-70% TDF; cellulose, hemicellulose, lignin, pectin (12-20%)</td>
<td>β-carotene</td>
<td>Larrauri, Rupérez, Borroto and Saura-Calixto (1996), Berardini, Fezer, et al. (2005), Berardini, Knödler, et al. (2005), Ajila et al. (2007), Vergara-Valencia et al. (2007), Martínez et al. (2012)</td>
</tr>
<tr>
<td>Mango seed kernels</td>
<td>Tannins, gallic acid, coumarin, caffeic acid, vanillin, mangiferin, ferulic acid, cinnamic acid, ellagic acid, galocatechin, acylated cyaniding, β-Sitosterol, δ-Avenasterol Campesterol, Stigmasterol α-Tocopherol, γ-Tocopherol</td>
<td>12% Oil (oleic acid, linoleic acid)</td>
<td></td>
<td>Arogba (2000), Puravankara et al. (2000), Abdalla et al. (2007), Barreto et al. (2008), Maisuthisakul and Gordon (2009)</td>
</tr>
<tr>
<td>Mangosteen rind</td>
<td>Tannins, xanthones (α-mangostin, β-mangostin, 3-isomangostin, 9-hydroxy calabaxanthone, gartanin, and 8-desoxygartanin), anthocyanins, proanthocyanidins, catechin</td>
<td></td>
<td></td>
<td>Jung et al. (2006), Fu et al. (2007), Ji et al. (2007), Zdernowski et al. (2009), Wittenauer et al. (2012),</td>
</tr>
<tr>
<td>Mangosteen seed</td>
<td></td>
<td>21% Unsaturated fatty acids (stearic acid, oleic acid, linoleic acid, gadoleic acid, and eicosadienoic acid)</td>
<td></td>
<td>Hawkins and Kridi (1998), Ajayi et al. (2007)</td>
</tr>
<tr>
<td>Passion fruit peel</td>
<td>Phenolic acids, Flavonoids</td>
<td>70-80% TDF (Cellulose, hemicellulose, pectic substances)</td>
<td></td>
<td>Silva et al. (2008), Kliemann et al. (2009), Martínez et al. (2012), López-Vargas et al. (2013)</td>
</tr>
<tr>
<td>Solid fruit waste</td>
<td>Phenolic compounds</td>
<td>Dietary fibre</td>
<td>Others bioactive components</td>
<td>References</td>
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<td>------------------------</td>
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</tr>
<tr>
<td>Passion fruit seed</td>
<td>Tocopherols</td>
<td>50% TDF (cellulose, pectic substances, hemicellulose)</td>
<td>30% Oil (linoleic acid, oleic acid)</td>
<td>Chau and Huang (2004), Malacrida and Jorge (2012), López-Vargas et al. (2013)</td>
</tr>
<tr>
<td>Blackcurrant pomace</td>
<td>Delphinidin-3-O-glucoside, delphinidin-3-O-rutinoside, cyanidin-3-O-glucoside, cyanidin-3-O-rutinoside</td>
<td>Cellulose, hemicellulose, pectin (2.7%), lignin</td>
<td></td>
<td>Kapasakalidis et al. (2006), Sójka et al. (2009), Holtung et al. (2011)</td>
</tr>
<tr>
<td>Sour cherry pomace</td>
<td>Neochlorogenic acid, 3-p-coumaroylquinic acid, chlorogenic acid, quercetin glucoside and rutinoside, kaempferol-rutinoside, isorhamnetin-rutinoside, quercetin, kaempferol, isorhamnetin, anthocyanins</td>
<td>Cellulose, hemicellulose, pectin (1.5%), lignin</td>
<td></td>
<td>Nawirska and Kwaśniewska (2005), Kołodziejczyk et al. (2013)</td>
</tr>
</tbody>
</table>
Isolation, quantification, and characterisation of phenolic compounds in fruit solid wastes have been heavily studied as they are present in all types and parts of fruits wastes (Table 2). Flavonoids and phenolic acids are the most common classes of phenolic compounds present. The profiles of these substances from different cultivars and fruit sources, can widely differ, both in terms of components and concentrations. For instance, Wolfe et al. (2003) demonstrated that phenolic compounds were most localised in apple peel. Apple seeds contain smaller range of phenolic compounds than the skin with phloridzin as major component (80-90%), (Lu and Foo 1998, Fromm et al. 2012). Variation in phenolic profiles is also evident in citrus. Lemon seed mainly contains high amounts of eriocitrin and hesperidin, while the peel is rich in neoeiocitrin, naringin and neohesperidin (Bocco et al. 1998). Hesperidin is the most abundant flavonoid in Valencia, Navel, Temple and Ambersweet orange peels (Manthey and Grohmann 1996) and naringin is the most abundant flavonoid in grapefruit peel (Wu et al. 2007). Grape pomace is rich in anthocyanins, catechins, procyanidins, flavonol glycosides, phenolic acids (Rodríguez Montealegre et al. 2006). The phenolic compounds in grape seeds are essentially all flavonoids, particularly, flavan-3-ol. Grape skin is rich in resveratrol. Pomegranate peel contains higher phenolic compounds, especially phenolic acids, than the pulp (Li, Guo, et al. 2006). Mangosteen peels are rich in tannins and anthocyanins (Wittenauer et al. 2012). Detail of selected reports regarding phenolic compounds in specific part and sources of fruit juice wastes is included in Table 2.

**Dietary fibre**

The relationship between dietary fibre and health has long been established (Buttriss and Stokes 2008). The beneficial physiological effects in humans include decreasing intestinal transit time and increasing faecal bulk fermentable h colonic microflora, reducing cholesterol levels in the blood, reducing insulin responses (Laurentin et al. 2003). Dietary fibre can also impart some functional properties which can improve food characteristics, such as increase water holding capacity, oil holding capacity, emulsification and gel formation (Belitz et al. 1999). Dietary fibre is a class of complex carbohydrates and can be divided into soluble and insoluble fibres. Fruit solid wastes are excellent sources of soluble
dietary fibre, such as pectin and gums, as well as insoluble dietary fibres, such as cellulose, hemicellulose, and lignin. Dietary fibre is associated to plant cell walls and tissues, therefore it is mostly located in peels, skins, pericarps, and stalks. High percentages of dietary fibre can be recovered pomaces of apples, grapes, citrus, pear, cherry, berries, passion fruit and mangos (Larrauri, Rupérez, Borroto and Saura-Calixto. 1996, Larrauri, Rupérez, Bravo and Saura-Calixto. 1996, Nawirska and Kwaśniewska 2005, Garau et al. 2007, Elleuch et al. 2011, Martínez et al. 2012, Amaya-Cruz et al. 2015).

Dietary fibre and pectin from citrus and apple peels have been produced in commercial scale (Rezzadori et al. 2012, Martí et al. 2014). Pectin yield depends on both technological factors and fruit physiology. In citrus, apart from extraction methods, intrinsic factors such as the type of citrus and the portion of waste considerably affected pectin yield (Widmer and Montanari 1994, Marín et al. 2007, Martí et al. 2014). Pectin recovered from apple pomace has superior gelling properties but was inferior in colour to citrus pectin. Removal of oxidised phenolic compounds improves the colour of apple pomace pectin without compromising its gelling properties (Schieber et al. 2003).

9.4 Extraction of bioactive compounds from fruit juice industry waste

Many factors need to be considered in order to achieve best results in the extraction of phenolic compounds and dietary fibre from fruit wastes. Understanding the nature of target compounds and of raw materials, as well as waste matrices is crucial to the success of the operation. Apart from the aforementioned factors, process types and operating parameters used in the recovery process are also important determinants of the yield and quality of the recovered compounds.

In general, recovery of target compounds from fruit wastes composes of (1) pre-treatment, (2) extraction, (3) isolation and purification, and (4) product formulation (Figure 3). The detail of overall general recovery process of bioactive compounds from food wastes have been previously described (Oreopoulou and Tzia 2007, Galanakis 2012).
Figure 3 A general flow diagram of recovery stages of bioactive compounds from fruit wastes. Solid lines represent the recovery stages of phenolic compounds, terpenes, carotenoids, and phytosterols; dash lines represent the recovery stages of dietary fibre and pectin.

**Phenolic compounds**

Important factors affecting the efficiency of the extraction of phenolics include their chemical nature, sample preparation (drying method, particle size, storage time and conditions), the extraction method employed (mode of extraction, extracting medium, solvent-to-solid ratio, contact time, temperature),
and presence of interfering substances (Pinelo et al. 2005, Naczk and Shahidi 2006, Valls et al. 2009, Çam and Aaby 2010, Candrawinata et al. 2014). The solubility of phenolic compounds is greatly affected by the polarity of solvent, extracting conditions, degree of polymerization of phenolic compounds, as well as interaction of phenolic compounds with other food constituents and formation of insoluble complexes (Naczk and Shahidi 2004). Phenolic extracts of fruit wastes are therefore always a mixture of different classes of phenolic compounds that are soluble in the extraction system applied. Conventional extractions result in dilute extracts, therefore concentration of the extracts may be required (Galanakis 2012).

The method chosen to dry fruit wastes prior to extraction can considerably affect the yield. Comparison studies of the effects of different drying methods on the yield of phenolic compounds extracted from grape skins (oven drying and freeze-drying; de Torres et al. 2010), apple pomace (vacuum-drying, oven drying, and freeze-drying; Lavelli and Corti 2011), mango peel and kernel (Freeze-drying, vacuum-drying, oven-drying, and infra-red-drying; Sogi et al. 2013) have been reported. In all studies, freeze-drying was less aggressive than thermal drying methods, especially to anthocyanins and anthocyanidins, and it was possible to maintain high antioxidant activity in the fruit matrices. It should be noted that, although freeze-drying is found to be gentler than thermal drying methods, losses of phenolic compounds are still observed (Paes et al. 2014). In the sample preparation step, if desired, unwanted phenolic and non-phenolic substances such as waxes, fats, terpenes and chlorophylls can be removed by washing off with nonpolar solvent (such as hexane) before extraction of target compounds (Huber and Rupasinghe 2009).

There is no standard or completely satisfactory method to extract phenolic compounds in fruit wastes as the method suitable for one material may not be suitable for others. Effective solvents generally used for extracting phenolic compounds from fruit wastes are ethyl acetate, ethyl ether, ethanol, acetone, methanol, including their aqueous mixtures (Ignat et al. 2011, O'Shea et al. 2012). Nonpolar solvents (hexane, petroleum ether) are suitable for extraction of tocopherols and certain phenolic terpenes (Oreopoulou and Tzia 2007). In solvent extraction, interference compounds, such as sugars and organic acids, are generally co-eluted into the extract. Removal of these compounds can be
achieved by passing the crude extract through C18 solid phase extraction prior to separation of phenolic components or antioxidant activity determination (Li, Smith, et al. 2006, Reis et al. 2012). Methanol is found to be a highly effective solvent especially for anthocyanin extraction (Kapasakalidis et al. 2006). (Wijngaard et al. 2009, Wijngaard and Brunton 2010) also reported methanol was the most effective solvent in comparison to ethanol and acetone on apple pomace phenolic yield and antioxidant activity.

Although some solvents are found to be highly effective, with the growing concerns on safety issues arising from their toxicity, they become less prefer option. In addition, some solvents potentially create health and safety challenges for production and impart harmful impurities into the phenolic extracts, especially if the extracts are intended for food applications. Due to this, investigation of ‘green’ extraction using water, solely or in high proportion, as extracting medium has been explored. Experimental results suggested that water, including diluted acidic solutions and buffers, are not as effective as organic solvents, but can be an acceptable extracting medium. Pinelo et al. (2005) compared the extraction efficiencies of solvents (ethanol, methanol, water), together with other operating parameters (time, temperature, and solvent-to-solid ratio) in grape pomace. The authors reported that, regardless of the solvent used, the highest yields of phenolic were obtained from the conditions with the highest temperature (50 °C), extracting time (90 min), and solvent-to-solid ratio (5 mL/g), in the range studied. Water was able to recover approximately 60% of phenolics relatively to that amount obtained from methanol. More recent studies showed that the efficiency of water as extracting medium can be further improved by increasing extracting temperature together with optimized solvent-to-solid ratio and extraction time in grape pomace (Çam and Aaby 2010), and apple pomace (Candrawinata et al. 2014). In addition to using water, solvents and their aqueous solutions, acidified extracting media can also improve yields, especially for anthocyanins. Weak organic acids (formic acid, acetic acid, citric acid, and tartaric acid), and low concentrations of strong acids (trifluoroacetic acid, hydrochloric acid), are beneficial for extracting anthocyanins (Revilla et al. 1998, Ju and Howard 2003).
Table 3 Extraction, separation, and characterization of phenolic compounds in selected fruit wastes

<table>
<thead>
<tr>
<th>Fruit Waste</th>
<th>Sample Preparation and Extraction Conditions</th>
<th>Separation / Characterization Method</th>
<th>Major Compounds Identified</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peach peel</td>
<td>Frozen; solvent: 80% aqueous methanol</td>
<td>LC-DAD; C18; Mobile phase: 50 mM ammonium phosphate, pH 2.6 (A), 80% acetonitrile and 20% buffer A (B), 200 mM orthophosphoric acid, pH 1.5 (C); Detection: 316 nm (hydroxycinnamates), 520 nm (anthocyanins), 280 nm (flavan-3-ols), 365 nm (flavanols)</td>
<td>Chlorogenic acid, procyanidin, catechin, isoquercetin B1, neochlorogenic acid, malvin, rutin</td>
<td>Chang et al. (2000)</td>
</tr>
<tr>
<td>Peach peel (yellow-, white-fleshed), Nectarine Peel (yellow-, white-fleshed), Plum peel</td>
<td>Frozen; solvent: water/methanol 2:8 containing 2 mM NaF (5g) was Solid-to-solvent ratio: 5:10 g/mL</td>
<td>HPLC-DAD/ESI-MS; C18; Mobile phase: 95% water + 5% methanol (A), 88% water + 12% methanol (B), 20% water + 80% methanol (C), methanol (D); Detection: 280, 340, 510 nm</td>
<td>Chlorogenic acid, catechin epicatechin, neochlorogenic acid, procyanidin B1, rutin, cyanidin 3-rutinoside</td>
<td>Tomás-Barberán et al. (2001)</td>
</tr>
<tr>
<td>Mango peel, Mango kernel</td>
<td>Freeze dried, defatted; solvent: methanol; temperature: RT</td>
<td>LC-DAD/ESI-MS; C18; Mobile phase: 2% acetic acid in water (A), methanol (B); Detection: 278, 340 nm</td>
<td>Peel: Penta-O-galloyl-glucoside, methyl gallate, mangiferin, tetra-O-galloyl-glucoside, maclurin di-O-galloyl-glucoside, isoquercitrin</td>
<td>Barreto et al. (2008)</td>
</tr>
<tr>
<td>Grape cane</td>
<td>Dried, ground; solvent: aqueous ethanol (36-80% v/v); temperature: 30-70°C; solvent-to-solid ratio: 50-90:1 mL/g</td>
<td>LC-DAD; C18; Mobile phase: 50 mM phosphoric acid (A), methanol (B); Detection: 280 nm</td>
<td>trans-resveratrol equivalent compounds</td>
<td>Karacabey and Mazza (2010)</td>
</tr>
</tbody>
</table>
Table 3 Extraction, separation, and characterization of phenolic compounds in selected fruit wastes (continued)

<table>
<thead>
<tr>
<th>Fruit Waste</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Pomegranate peel and mesocarp</td>
<td>Lyophilized; Methanol</td>
<td>LC-DAD/ESI-MS; C18 column; Anthocyanins – Mobile phase: 5% v/v formic acid in water (A), water, formic acid and methanol (10/10/80, v/v/v, B); Detection: 520 nm</td>
<td>Peel: anthocyanins (Cyadinin-3,5-diglucoside, pelargonidin-3,5-giglucoside), ellagitannins (granatin B, castalagin der, galloyl-HHDP-hex, bis-HHDP-hex), gallic acid Mesocarp: ellagitannins (galloyl-HHDP-gluconic acid, granatin B ellagic acid der, digalloyl-HHDP-gluconic acid)</td>
<td>Fischer et al. (2011)</td>
</tr>
<tr>
<td>Pomegranate peel</td>
<td>Air drying, ground; 80% methanol</td>
<td>MALDI-TOF MS; 337 nm pulsed nitrogen laser, polarity-positive (alternatively negative), flight path-linear, 20 kV acceleration voltages, 100–150 pulses per spectrum</td>
<td>flavonoid tetramers, pentagalloyl glucose, hydrolyzable tannins, ellagitannins</td>
<td>Saad et al. (2012)</td>
</tr>
<tr>
<td>Apple seeds</td>
<td>Lyophilized, ground, defatted; Aqueous acetone (30:70 v/v) followed by liquid–liquid extraction with ethyl acetate</td>
<td>LC-DAD/ESI-MS; C18; Mobile phase: 2% acetic acid in water (A), 0.5% acetic acid in water and methanol (30:70, B); Detection: 280 nm (dihydrochalcones, flavanols), 320 nm (hydroxycinnamic acids), 370 nm (flavonols)</td>
<td>Phloridzin, epicatechin, catechin</td>
<td>Fromm et al. (2012)</td>
</tr>
<tr>
<td>Sour cherry pomace</td>
<td>Water (70°C), followed by extract purification on Amberlite XAD-7HP column</td>
<td>LC-DAD/ESI-MS; C18 column; Mobile phase: 10% v/v formic acid in water (A), 50:40:10 v/v/v acetonitrile:water:formic acid (B); Detection: 320 nm (hydroxycinnamic acids), 360 nm (quercetin, kaempferol, isorhamnetin glycosides and their aglycones), 520 nm (anthocyanins) LC-UV; C18 column; 280nm (flavanols)</td>
<td>Cyanidin-glucoside-rutinoside, chlorogenic acid, neochlorogenic acid, p-coumaroylquinic acid, quercetin, kaempferol, isorhamnetin glycosides</td>
<td>Kołodzieczyk et al. (2013)</td>
</tr>
<tr>
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<tr>
<td>Apple pomace</td>
<td>Freeze drying, ground; PLE: 25-75% aqueous ethanol. Temperature: 160-193°C. Pressure: 10.3 MP. Extraction time: 5 min</td>
<td>LC-DAD; C18; Mobile phase: acetic acid in 2mM sodium acetate (pH 2.55, v/v, A), 100% acetonitrile (B); Detection: 280 nm (hydroxybenzoic acids, dihydrochalcones, procyanidins, flavonoids), 320 nm (hydroxycinnamic acid derivatives), 360 nm (flavonols)</td>
<td>Chlorogenic acid, flavonols, and phloretin glucosides.</td>
<td>Wijngaard et al. (2009)</td>
</tr>
<tr>
<td>Pomegranate peels</td>
<td>Sun drying, ground; PLE: water. Pressure: 102.1 atm. Extraction time: 5 min</td>
<td>LC-DAD; C18; Mobile phase: water/acetic acid (98:2, v/v, A), methanol (B); Detection: 260, 280, 320, 360, and 378 nm. Spectrophotometry method for tannins</td>
<td>Punicalagin B, punicalagin A, ellagic acid derivatives, gallic acid Condensed tannins, hydrolysed tannins</td>
<td>Çam and Hişıl (2010)</td>
</tr>
<tr>
<td>Grape seeds</td>
<td>Air drying, ground; SC-CO2 with 5-20% ethanol. Temperature: 30, 50°C. Pressure: 25–30 MPa. Extraction time: 60 min</td>
<td>LC-DAD; C18; Mobile phase: 2% acetic acid in water (A),100% acetonitrile (B); Detection: 280 nm</td>
<td>Gallic acid, epigallocatechin, epigallocatechingallate, catechin, epicatechin, epicatechingallate:</td>
<td>Yilmaz et al. (2011)</td>
</tr>
<tr>
<td>Pomegranate seeds</td>
<td>Ground, defatted by SC-CO2 (37.9 MPa, 47.0 °C); PLE: water. Pressure: 6.0 MPa. Temperature: 80–280 °C. Extraction time: 15–120min</td>
<td>LC–ABTS•+; C18; Mobile phase: 0.2% v/v formic acid (A), methanol (B); Detection: 280 nm</td>
<td>Caffeic acid derivative, catechin, kaempferol 3-O-rutinoside,</td>
<td>He et al. (2012)</td>
</tr>
<tr>
<td>Fruit Waste</td>
<td>Sample Preparation and Extraction Conditions</td>
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<tr>
<td>Apple pomace</td>
<td>Lyophilized. Ground SC-CO2 + 25%ethanol (25 MPa, 50 °C)</td>
<td>LC-DAD/ESI/ACPI-MS; C18; Mobile phase: 0.1% formic acid (A), acetonitrile (B); Detection: 280 nm</td>
<td>Quercetin-3-O-xiloside, Quercetin-3-O-rhamnoside Quercetin-3-O-arabinoside Quercetin-3-O-glucoside Epicatechin, Quercetin-3-O-galactoside Phloridzin, Catechin, Chlorogenic acid</td>
<td>Garcia-Mendoza et al. (2015)</td>
</tr>
<tr>
<td>Blackcurrant pomace</td>
<td>Oven dried, ground; UAE Solvent: methanol:water:formic acid (50:48:2 v/v/v). Solid:solvent ratio: 0.5:5 g/mL</td>
<td>LC-DAD/ESI-MS; C18 column; Mobile phase: 10% v/v formic acid in water (A), 50:40:10 v/v acetonitrile:water:formic acid (B); Detection: 320 nm (hydroxycinnamic acids), 360 nm (quercetin and myricetin), 520 nm (anthocyanins)</td>
<td>Delphinidin-3-rutinoside, Delphinidin-3-glucoside, Cyanidin-3-rutinoside, Cyanidin-3-glucoside, Myricetin, Quercetin, Kaempferol</td>
<td>Sójka et al. (2009)</td>
</tr>
<tr>
<td>Blackcurrant seeds</td>
<td>Oven dried, ground; UAE Solvent: Ethanol</td>
<td>LC-UV; C8; Mobile phase: 0.1% acetic acid (A), 10% acetonitrile (B); Detection: 280 nm</td>
<td>Procyanidins, cinnamic acid, chlorogenic acid, caffeic acid, syringic acid</td>
<td>Bai et al. (2010)</td>
</tr>
<tr>
<td>Apple pomace</td>
<td>Oven-dried (60 °C); Reflux, MAE, UAE Solvent: Ethanol</td>
<td>LC-DAD/ESI-MS, C¹³ NMR; C18; Mobile phase: 40% methanol (A), 100% methanol (B); Detection: 284 and 332 nm (flavonoids).</td>
<td>Hesperidin, narirutin, nobiletin</td>
<td>Inoue et al. (2010)</td>
</tr>
<tr>
<td>Citrus peel</td>
<td>Ground; Conventional extraction: 0-100% ethanol, and DMSO:methanol,1:1, v/v MAE: 70% ethanol</td>
<td>LC-DAD/ESI-MS; C¹³ NMR; C18; Mobile phase: 40% methanol (A), 100% methanol (B); Detection: 284 and 332 nm (flavonoids).</td>
<td>Hesperidin, narirutin, nobiletin</td>
<td>Inoue et al. (2010)</td>
</tr>
<tr>
<td>Orange peel</td>
<td>UAE; 20-80% v/v Ethanol</td>
<td>LC-DAD; C18; Mobile phase: 0.5% acetic acid (A), 100% acetonitrile (B); Detection: 280 nm</td>
<td>Naringin, hesperidin</td>
<td>Khan et al. (2010)</td>
</tr>
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</table>
Table 3 Extraction, separation, and characterization of phenolic compounds in selected fruit wastes (continued)

<table>
<thead>
<tr>
<th>Fruit Waste</th>
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<th>Major Compounds Identified</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Citrus peels</td>
<td>Fresh and dried; UAE at 60 kHz, Peel moisture content: 0%, 75%; Time: 30, 90 min solid/water ratio: 1/10 g/mL</td>
<td>LC–DAD/ESI–MS; C18; Mobile phase: 0.1% formic acid (A), acetonitrile (B); Detection: 280 nm</td>
<td>Hesperidin, neohesperidin, diosmin, nobiletin, tangderitin</td>
<td>Londoño-Londoño et al. (2010)</td>
</tr>
<tr>
<td>Grape skins</td>
<td>MAE Solvent: 50–80% MeOH Temperature: 50–100 oC), Time: 5–20 min Microwave power: 100–500 W) Solid:solvent ratio: 1:12.5–1:25 g/mL</td>
<td>LC-DAD; C18; Mobile phase: 5% formic acid (A), methanol (B); Detection: 520 nm</td>
<td>Malvidin 3-glucoside, peonidin 3-glucoside, malvidin 3-acetylglucoside, petunidin 3-glucoside, malvidin 3-p-coumaroylglucoside, delphinidin 3-glucoside, malvidin 3-caffeoylglucoside</td>
<td>Liazid et al. (2011)</td>
</tr>
<tr>
<td>Grape seeds</td>
<td>Air drying, ground; MA aqueous two-phase extraction Solvents: 24% to 34% (w/w) acetone and 14% to 22% (w/w) ammonium citrate Time: 2min</td>
<td>LC-UV; C18; Mobile phase: 0.3% phosphoric acid (A), methanol (B); Detection: 280 nm</td>
<td>Catechin, gallic acid, epicatechin, trans-resveratrol, quercetin</td>
<td>Dang et al. (2014)</td>
</tr>
<tr>
<td>Lime pomace</td>
<td>Freeze-dried, tray-tried (60, 90 and 120 °C); UAE: 80% Methanol at RT in ultrasonic bath followed by methanol/H₂SO₄ hydrolysis for non-extractable phenolics</td>
<td>LC-DAD; C18; Mobile phase: 6% acetic acid in 2 mM sodium acetate buffer (pH 2.55, v/v, A), acetonitrile (B); Detection: 260 nm (hydroxybenzoic acids, quercetin, rutin), 280 nm (flavans and flavanones), 320 nm (hydroxycinnamic acids, stilbenes), 360 nm (miricetin and kaempherol)</td>
<td>Hesperidin Eriocitrin Naringin Naringenin p-Coumaric Benzoic Ellagic Catechin</td>
<td>Esparza-Martínez et al. (2016)</td>
</tr>
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</table>
Besides the conventional solvent extraction, other techniques have been introduced in an attempt to improve the extraction process to obtain extracts with higher yield and functional activities. Over the last decade, applications of compressed fluid extraction (pressurized liquids, supercritical fluids), microwave, sonication, (as pre-treatments or sole extraction methods) have become strong candidates of choice. These technologies have proven beneficial by improving yield / biological activities of target compounds from fruit wastes, more economical to run, and, highly acceptable as green processes when applied with a carefully chosen extracting media (Galanakis 2012, Galanakis 2013).

Compressed fluid extraction must operate under medium-to-high pressures. Extraction methods using this approach include pressurized liquid extraction (PLE), subcritical water extraction (SWE), and supercritical fluid extraction (SFE). Operating principle of PLE is to use liquids (extraction media) at temperatures above their normal boiling points and under enough pressures to keep the extracting fluid in the liquid state. When applied PLE with water as extracting medium, the process is called subcritical water extraction (SWE). PLE enables rapid extraction (3 - 20 min) of analytes in a closed and inert environment, under high pressures (3.3 - 20.3 MPa) and temperatures (40 - 200 °C) (Richter et al. 1996). The most important operating parameter in PLE applications is temperature. In general, recovery of higher bioactive yields at higher amounts at higher temperature have been observed but simultaneously too high temperature might be detrimental to biological activities of the extracts. Šťavíková et al. (2011) used pressurized water (15 MPa) to extract anthocyanins from grape skins and found that the recovery of anthocyanins as well as radical-scavenging abilities of the extracts were dependent on extraction temperature (up to 80°C). This trend, however, is not observed when using methanol or ethanol pressurized under the same pressure as optimal temperature was found to be 40 °C (Polovka et al. 2010). Therefore, this parameter should be studied and selected for each type of matrix or bioactive being extracted. Other parameters (e.g., pressure and time) are also important but pose a less critical effect (Herrero et al. 2013).

Supercritical fluid extraction (SFE) is another application of compressed fluid extraction. SFE operates at temperature and pressure close to the critical point of the solvent used. Based on this operating principle, the most utilised critical fluid has been carbon dioxide (CO₂) because of its low
critical temperature and pressure (31.1 °C, and 7.4 MPa, respectively; Hawthorne 1990). Low operating temperature is beneficial to extraction of phenolic compounds which are thermolabile. Carbon dioxide has low toxicity and is safe for food application and SC-CO$_2$ is considered safe and green (Reverchon and De Marco 2006). In SC-CO$_2$ system, other solvents are not generally necessary, although the presence of co-solvents (such as methanol, ethanol, water) may be beneficial, especially in the case of anthocyanin extraction (Bleve et al. 2008, Wijngaard et al. 2012). This is because CO$_2$ has low polarity, and small quantity of co-solvents (generally lower than 15%) are commonly required to modify the effectiveness of CO$_2$ in extracting more polar compounds. Key operating parameters needs to be optimized in SFE applications include sample particle size, temperature, pressure, time, co-solvents, solvent-to-solid ratio (da Silva et al. 2016).

Extraction performance of conventional solvent extraction, PLE, and SFE has been compared (Paes et al. 2014). Anthocyanin extraction from blueberry pomace using conventional solvent extraction (methanol, ethanol, acetone), PLE (acidified water, ethanol, 50% v/v aqueous ethanol, 50% v/v ethanol in acidified water, acetone), and SC-CO$_2$ have been investigated. Among the methods and conditions tested, the authors reported that PLE and SFE was effective on the extraction of phenolics, antioxidants, and anthocyanins from blueberry wastes, particularly PLE with water and/or ethanol, and SC-CO$_2$ with 5% water and 5% ethanol as co-solvents. Interestingly, Garcia-Mendoza et al. (2015) combined SC-CO$_2$ and PLE (ethanol) into sequential extraction steps to extract phenolic compounds form mango peel. The results showed the extraction yield was improved as non-polar phenolic compounds were recovered by SC-CO$_2$ in the first stage while polar phenolic components were extracted by pressurized ethanol during the second stage.

Microwave-assisted extraction has been reported to accelerate extraction time and improve phenolic yields (Inoue et al. 2010). Microwaves are non-ionizing radiation with frequencies between 300 MHz and 300 GHz. Microwaves can interact with polar solvent (such as ethanol, methanol, water) and heat the solvent rapidly, causing moisture loss in the cells. The steam generated then swells and penetrates the sample matrix, resulting in cell walls disruption and fast migration of phenolics into the solvent (Wang and Weller 2006). Important operating parameters include type of solvent, solvent-to-
solid ratio, microwave energy, extracting time, and temperature (Hayat et al. 2010, Zhang et al. 2011, Rezaei et al. 2013). MAE process conditions for phenolic recovery have been investigated in a number of fruits solid wastes (Table 3).

Ultrasound-assisted extraction is one of the emerging extraction techniques offers many advantages, such as rapid, reproducible, economical, and clean. Ultrasound with frequencies higher than 20 Hz creates transient cavitation (bubbles) to the sample matrix, leading to cell wall disruption and diffusion of phenolics into the solvent without significantly increase the temperature (Soria and Villamiel 2010). Indeed, optimization of operating parameters (temperature, solvent system, sonication power, sonication time, solvent-to-solid ratio, particle size) needs to be carried out to achieve yield improvement. Optimization of operating factors such as particle size, the extraction solvent, solid/solvent ratio, temperature, extraction time, the electrical acoustic intensity, liquid height and duty cycle have been studied in various types of fruit wastes. The effect of ultrasound on phenolic extraction has been tested in various studies and enhanced extraction has been observed (Khan et al. 2010, Virot et al. 2010, Pingret et al. 2012, Dahmoune et al. 2013).

Combination of the aforementioned extraction techniques in order to achieve better results has also been investigated. Applying ultrasound during SC-CO₂ with water as a co-solvent was found to remarkably increase extraction rate and yields of phenolics, anthocyanins, as well as the antioxidant activity of the extracts obtained from blackberry bagasse. Using ethanol as a co-solvent also exerted positive influence on the extraction of anthocyanins, but the effect was much less pronounced than water (Pasquel Reátegui et al. 2014).

Comparison of extraction performance of several extraction methods (conventional solvent extraction (methanol and ethanol), UAE, MAE, and high pressure and temperature extraction (HPTE; water)) to obtain phenolic compounds from grape seeds and skins has been reported (Casazza et al. 2010). The authors reported that HPTE provided the highest content of total phenolics both for seeds and skins, while MAE retained the highest antiradical power. Prolonged extraction times (over 30 min) was not necessarily beneficial because although the amount of total polyphenols increased, the amount of flavonoids and the antiradical power decreased.
Emerging extraction techniques have recently been implemented for recovery of phenolic compound from fruit wastes. Application of electrotechnology, such as pulsed electric field (PEF), and high voltage electrical discharge (HVED), has gain increased interest. Both techniques are non-thermal processes, which are highly beneficial for recovery of heat-sensitive compounds. PEF and HVED have been shown to be promising for intracellular extraction from by-products (Luengo et al. 2013, Boussetta and Vorobiev 2014).

PEF uses strong electric field to provoke pre formation on the cell structure. This electroporation (or electropermeabilization) facilitates the release of target compounds from the fruit matrices (Wijngaard et al. 2012). PEF-assisted extraction generally involves direct electric pulsed of high voltage are applied (upto 40 kV) for short duration (less than 10 ms) at a repeated pulse (frequency), resulting in high electric field strength (1–10 kV/cm). Efficiency of PEF-assisted extraction is dependent on the PEF system configuration and extraction parameters. Similar to other methods, extraction temperature, sample particle size, solvent system and concentration, are important factors determining the extraction performance. Enhanced PEF extraction yields of phenolic compounds from orange peels (Luengo et al. 2013), anthocyanins from blueberry pomace (Zhou et al. 2015), phenolic compounds and anthocyanins from raspberry pomace (Lamanaukas et al. 2016) have been reported. In another study by Medina-Meza and Barbosa-Cánovas (2015), PEF offered enhanced anthocyanin yield from grape peel but the yield was not impressive when the same PEF conditions was applied to plum peels.

HVED works based on chemical reactions and physical processes. HVED have electrical and mechanical effects on the product caused by shock waves. This technique introduces energy directly into an aqueous solution through a plasma channel formed by a high-current/high-voltage electrical discharge between two submerged electrodes. Large range of current ($10^3$ - $10^4$ A), voltage ($10^3$–$10^4$ V) and frequency ($10^{-2}$ - $10^{-3}$ Hz) are typically applied (Boussetta and Vorobiev 2014). Extraction parameters affecting the extraction yield include solvent system, inter-electrode space, energy input, liquid-to-solid ratio and temperature. HVED has been satisfactorily used to extract phenolic compounds from grape pomace (Boussetta, Lanoisellé, et al. 2009, Boussetta, Lebovka, et al. 2009, Boussetta et al. 2011), grape seeds (Liu et al. 2011).
HVED has been reported to be more efficient than PEF in the extraction of phenolic compounds from grape skins (Boussetta, Lebovka, et al. 2009), grape pomace (Barba et al. 2015), and mango peel (Parniakov et al. 2016). It is rather interesting that PEF efficacy can be markedly improved when the treatment is performed at 50 °C and in the presence of ethanol (Boussetta et al. 2012) or with a supplementary aqueous extraction after PEF treatment (Parniakov et al. 2016).

**Dietary Fibre**

Fruit wastes are a rich sources of dietary fibre (DF). Cellulose, hemicelluloses, pectin, and lignin are typical fibre components found. The constituents are divided into soluble dietary fibre (SDF, i.e. pectin) and insoluble dietary fibre (IDF, i.e. cellulose, most hemicelluloses, lignin). They provide various functional effects beneficial to the human health, as well as functional properties in food processing and food formulation without offering nutritional value. Upon hydration, soluble fibres are able to form a gel or a network, while insoluble fibres are able to absorb large amount of water (up to 20 times their weight in water) and expand into bulky materials (Thebaudin et al. 1997, Figuerola et al. 2005, Nawirska and Kwaśniewska 2005, O'Shea et al. 2012).

*High dietary fibre concentrate / powder* High dietary fibre products can be prepared directly from fruit wastes or, if desired, after the recovery of other bioactive compounds (Fig 2). The simplest preparation method is merely grinding of dried fruit wastes into fine particles. Conventional production of dietary fibre powder from fruit wastes involves a few mechanical steps, i.e. wet milling, washing, drying, and lastly dry milling (Oreopoulou and Tzia 2007). All the steps, although relatively simple, need to be optimized as they affect yield and characteristics of the obtained fibre (Larrauri 1999). An appropriate mean particle size from wet milling will ensure an adequate wash without holding too large amount of water which will make subsequent drying more difficult. In the washing step, washing time, water temperature, water-to-solid ratio are important parameters for maximizing removal of undesirable components (i.e. sugars), which will improve functionality and colour of the final product, and retain
desirable water-soluble components (i.e. soluble dietary fibre; Larrauri, Rupérez, Borroto and Saura-Calixto. 1996, Lario et al. 2004). Operating drying parameters, such as temperature, time and drying rate, affect the degradation, thus the yield, of target compounds (phenolic compounds, dietary fibres; Garau et al. 2007). Lastly, appropriate particle size from dry milling also needs to be determined as it affects the characteristics of the final products, such as water- and oil-holding capacity and suspension in water (Oreopoulou and Tzia 2007). Selected reports on extraction conditions of dietary fibre products from fruit wastes is shown in Table 4. Fruit wastes reported as good sources for dietary fibre recovery include pomaces of citrus, apple, pear, peach, passion fruit, mango, and pomegranate (Grigelmo-Miguel and Martín-Belloso 1998, Grigelmo-Miguel et al. 1999, Grigelmo-Miguel and Martín-Belloso 1999, Larrauri 1999, Lario et al. 2004, Figuerola et al. 2005, Viuda-Martos et al. 2012, Ajila and Prasada Rao 2013, López-Vargas et al. 2013).

Without any extraction step prior to fibre preparation, dietary fibres obtained directly from fruit wastes contain high amounts of bioactive compounds such as phenolic compounds, terpenes, carotenoids – depending on the fruit sources (Saura-Calixto 2010). Lime peel dietary fibre powder is found to have much stronger antioxidant activity than orange peel dietary fibre powder as it contains a broader range of phenolic components (caffeic acid, ferulic acids, naringin, hesperidin, myricetin, ellagic acid, quercetin, kaempferol; (Larrauri, Rupérez, Bravo and Saura-Calixto. 1996). Presence of phenolic compounds can cause discoloration of the final product. Applications of alkaline solution / ozone ultrasonic assisted extraction has been patented as a decolouration method to improve the product’s colour (Chen and Li 2013).

Dietary fibre with lower IDF/SDF ratio is considered of better quality and is more desirable as a food ingredient. The composition of polysaccharide constituents in dietary fibres depends on the sources of fruit wastes. Fibres from cherry and blackcurrant pomaces contain low amounts of pectin and amounts of lignin, thus have much higher IDF/SDF ratio than fibre from apple pomace (Nawirska and Kwaśniewska 2005).
### Table 4 Selected studies on preparation of dietary fibre products from fruit wastes

<table>
<thead>
<tr>
<th>Dietary Fibre Product</th>
<th>Fruit Waste</th>
<th>Extraction Conditions / Analysis method</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>fibre concentrate</td>
<td>Passion fruit seeds</td>
<td>Cleaned, finely ground to 0.5 mm size, defatted; Enzymatic-gravimetric method: AOAC method 991.43</td>
<td>Chau and Huang (2004)</td>
</tr>
<tr>
<td>customized functional fibre</td>
<td>Citrus – whole, peel, pulp (sour range, satsuma, grapefruit, sweet orange)</td>
<td>Scalded in a water bath Drying: Oven at 50 ± 5 °C, 24 h Dry mill: 0.2 mm; Enzymatic-gravimetric method: Prosky et al. (1988)</td>
<td>Marín et al. (2007)</td>
</tr>
<tr>
<td>high dietary fibre</td>
<td>Apple – parenchyma tissues, pomace</td>
<td>Frozen, ground, then precipitate either in 72% ethanol or HEPES buffer; Enzymatic-chemical method: uronic acid content</td>
<td>Sun-Waterhouse et al. (2008)</td>
</tr>
<tr>
<td>high dietary fibre powder</td>
<td>Lime pomace</td>
<td>Washing: water, 95 °C, 5 min Soaking: ethanol (95% v/v) Drying: Oven at 60 °C Dry mill: 38–63, 63–150, 150–250, 250–300 and 300–450 μm; Enzymatic-gravimetric method: AOAC method 991.43</td>
<td>Peerajit et al. (2012)</td>
</tr>
<tr>
<td>dietary fiber powder</td>
<td>Yellow passion fruit – pomace, albedo</td>
<td>Washing: water, 45 °C, 8 min Drying: Oven 60 °C, 24 h Dry milling: less than 0.417 mm; Enzymatic-gravimetric method: AOAC method 991.43</td>
<td>López-Vargas et al. (2013)</td>
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**Pectin** Pectin is a family of complex polysaccharides of α-(1→4) galacturonic acid present in the primary cell wall and middle lamella of the plant tissues. All pectins are characterized by a high content of galacturonic acid (GalA), and, according to the regulation of FAO and EU, pectin must contain at least 65% GalA (Rolin 2002). Conventionally, pectin from fruit wastes can be extracted by the use of...
mineral acids, usually hydrochloric or nitric acid. The extract is separated from the solid residue and pectin is precipitated by the addition of ethanol. The precipitated pectin is then purified by washing with acidified, alkaline, and finally neutral alcohol. Lastly, the product is dried to a desirable moisture content. Citrus peel and apple pomace have been used to produce pectin in industrial scale (Oreopoulou and Tzia 2007, Martí et al. 2011, O'Shea et al. 2012). However, other fruit wastes are found to yield high amount of pectin, such as peach pomace (Pagan and Ibarz 1999, Pagan et al. 1999), passion fruit peels (Silva et al. 2008, Kliemann et al. 2009, Kulkarni and Vijayanand 2010), and mango peels (Rehman et al. 2004, Berardini, Knödler, et al. 2005).

Several studies have shown that, apart from the source and type of fruit waste used as raw material, the yield and quality of the obtained pectin greatly affected by the extraction conditions (acid type and concentration, pH, extraction time; Virk and Sogi 2004, Faravash and Ashtiani 2007, Kliemann et al. 2009). In general, yield is improved by low pH and high temperature or long time of extraction. However, these extraction criteria adversely affect gelling quality of pectin (Aravantinos-Zafiris and Oreopoulou 1992, Pagan et al. 1999). Phenolic compounds should be removed before pectin extraction as they cause undesirable light brown colouring in the produced pectin, especially when drying under temperature higher than 60 °C. Removal of phenolic compounds can be achieved by conventional and non-conventional extraction methods described in the previous section. Alternatively, implementation of resin absorption can successfully separate phenolic compounds, which can be subsequently recoverable, from the raw materials (Schieber et al. 2003, Berardini, Knödler, et al. 2005).

Applications of MAE and UAE in pectin recovery from fruit wastes have demonstrated high potential because those techniques can shorten extraction time, reduce solvent consumption, and improve extraction yield and functional properties of the obtained pectins (Table 5). Bagherian et al. (2011) did a comparison study on pectin extraction from grapefruit peel using MAE, UAE, and conventional methods. The author reported that MAE provided highest pectin yield with the best characteristics within the shortest extraction time. The extraction yield was also further improved when UAE was applied as a pretreatment for MAE. Another more recent comparison study investigated the efficacies of four different methods (MAE, UAE, conventional extraction, enzymatic extraction) on
pectin extraction from apple pomace (Li et al. 2014). The results showed enzymatic extraction was the best extraction method in terms of improving yield and functionality of extracted pectin. Pectin yield obtained from UAE was slightly higher than that from MAE; however, MAE drastically reduced extraction time. In comparison to conventional extraction, all non-conventional methods studied gave pectins of higher yields and improved functionality at a shorter extraction time.

Table 5 Selected studies on preparation of pectin from fruit wastes

<table>
<thead>
<tr>
<th>Fruit Waste</th>
<th>Extraction Conditions</th>
<th>References</th>
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<tbody>
<tr>
<td>Orange albedo</td>
<td>MAE under pressure, pH 1-2 Temperature (max.): 195 °C Pressure (max.): 50 ± 2 psi Solid-to-solvent ratio: 1:25, 5:25 g/mL Microwave power: 630 W at 2450 MHz</td>
<td>Fishman et al. (1999)</td>
</tr>
<tr>
<td>Apple pomace</td>
<td>MAE, pH 1.22-1.78 Time: 10.6-17.4 min Solid:liquid ratio (w/v): 0.0333 - 0.0571 Microwave power: 320, 450, 580 W</td>
<td>Wang et al. (2007)</td>
</tr>
<tr>
<td>Orange peel</td>
<td>MAE, pH 1-2 Time: 60-180 s Solid-to-solvent ratio: 1:10-1:30 g/mL Microwave power: 160-480 W</td>
<td>Maran et al. (2013)</td>
</tr>
<tr>
<td>Passion fruit peel</td>
<td>MAE, pH 2 Acid: acetic, tartaric, nitric Time: 3-9 min Solid-to-solvent ratio: 1:25 g/mL</td>
<td>Seixas et al. (2014)</td>
</tr>
<tr>
<td>Pomegranate peel</td>
<td>UAE, pH 1-2 Temperature: 50-70°C Time: 12-25 min Solid-to-solvent ratio: 1:10-1:20 g/mL</td>
<td>Moorthy et al. (2015)</td>
</tr>
<tr>
<td>Grapefruit peel</td>
<td>UAE, pH 1.5 Power intensity: 10/18-14.26 W/cm² Sonication time: 20-40 min Temperature 60-80 °C</td>
<td>Wang et al. (2015)</td>
</tr>
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</table>
9.5 Isolation of bioactive compounds from fruit juice industry waste

Due to the complex nature of both fruit materials and bioactive compounds recovered from them, many analysis techniques have been explored and developed to isolate, quantify, and characterize these bioactive compounds. Each technique has its own advantages and limitations. Common methods for isolation / quantification / characterization of phenolic compound and dietary fibres are discussed below.

**Phenolic compounds** Isolation of phenolic compounds can be achieved by various methods. Spectrophotometric methods, such as Folin–Ciocalteu, DPPH, ABTS, TEAC, FRAP assay, have been widely used for determination of phenolic compounds extracted from fruit wastes. These assays are relatively simple to perform with low running cost (Ignat et al. 2011). Nevertheless they offer little information in terms of what polyphenols are in the sample. They are non-selective, therefore, overestimation from interference presence in the samples is one common drawbacks. Comparison of experimental data is generally difficult as they are not standardised.

Liquid chromatography is a better choice for separation and quantification of phenolic compounds in fruit wastes as it is more sensitive and compound-specific. In most cases in fruit waste phenolic studies, separation is achieved by reversed-phase C18 column with gradient elution. In general, a binary solvent system composed of an acidified water (dilute formic acid or acetic acid) and a less polar organic solvent (ethanol, methanol or acetonitrile) is used, but tertiary or quaternary solvent systems are also reported (Chang et al. 2000, Tomás-Barberán et al. 2001). The acidic additive in the mobile phase is necessary to suppress the ionisation of the phenolic hydroxyl groups to obtain sharper peaks and minimised peak tailing. UV-Vis photodiode array detector (DAD) is a suitable detection mode for monitoring and quantifying different classes of phenolic compounds. As mentioned previously, phenolic compounds in fruit wastes are always a mixture of different phenolic classes, with different maximum absorption. In general, phenolic acids are detected at 220–280 nm, flavones and flavonols at 350–365 nm and anthocyanins at 460–560 nm (Valls et al. 2009). DAD is able to scan light
spectra in the UV-Vis range, thus allows easier monitoring of any separated phenolic fractions. Sakakibara et al. (2003) developed LC-DAD method and made a library, comprising HPLC retention times and spectra of aglycons for 100 standard chemicals, for simultaneously determining all phenolic compounds in a wide range of food samples (vegetables, fruits, and teas). LC-DAD system has been reported to successfully separate and quantify anthocyanins, procyanidins, flavonones, flavonols, flavan-3-ols, flavones, and phenolic acids in various types of fruit wastes (Table 4).

Although LC-DAD has been reported to be able to satisfactorily separate and quantify phenolic compounds in fruit wastes, it also presents limitations. As phenolic compounds are present ubiquitously in fruit wastes and their structure can be extremely complex, standards of only certain known compounds are available, which is one major limitation of the use of LC systems. Mass spectrometry (MS) is an analytical technique that is used for elucidating the chemical structures of molecules and plays a very important role for the analysis of polyphenolic compounds. MS structural elucidation is based on ionisation of chemical compounds to generate charged molecules or molecule fragments and measuring their mass-to-charge ratios (Sparkman 2000). To date, LC coupled with MS (LC-MS) technique is the most powerful and effective method for separation and characterization of complex phenolic structures such as procyanidins, proanthocyanidins, prodelphinidins, and tannins, including elucidation of speculated or hypothesised structures (Flamini 2003). Among the methods used for the determination of phenolics in crude plant extracts, liquid chromatography coupled with electrospray ionization (ESI) source has been widely used as it is a powerful tool owing to the soft ionization, which facilitates the analysis of this polar, non-volatile, and thermally labile class of compounds (Table 4). Matrix-assisted-laser-desorption-ionisation-time-of-flight (MALDI–TOF) techniques have also been used to characterize phenolic compounds in pomegranate peel (Saad et al. 2012). Sánchez-Rabaneda et al. (2004) employed LC/MS/MS and successfully identified 60 phenolic compounds from apple pomace, of which 23 components were described for the first time. The main advantages of MS/MS include exclusion of interferences and verification of the structures of the different compounds present in an extract.
Dietary fibre Isolation and quantification of soluble (pectin), insoluble (lignin, cellulose, hemicellulose), and total dietary fibre (TDF) in dietary fibre products prepared from fruit wastes can be achieved by various approaches. One of the easiest approaches used in fruit waste studies are non-enzymatic-gravimetric methods (Lario et al. 2004, Martí et al. 2011). Dietary fibre is characterized as crude fibre, acid detergent fibre (cellulose, lignin and acid insoluble hemicellulose), and neutral detergent fibre (neutral detergent insoluble hemicellulose, lignin, and cellulose). This approach, however, does not measure soluble dietary fibre, leading to underestimation of dietary fibre in the samples (Southgate et al. 1978).

In many studies, dietary fibre in fruit wastes was determined using the AOAC Prosky method (AOAC method 985.29), which is enzymatic-gravimetric based (Table 4). General procedure involves removal of starch and protein by the treatment of enzymes (α-amylase, protease, and amylglucosidase), followed by alcohol precipitation, filtration, and weighing of dietary fibre. Correction of protein and ash residue is also taken into account to prevent overestimation of dietary fibre (Prosky et al. 1984). Variation of the classical Prosky method has later been proposed and adopted as a standard method (AOAC method 991.43, Lee et al. 1992).

Apart from the enzymatic-gravimetric method, enzymatic-chemical method is also used in the determination of dietary fibre in fruit wastes (Larrauri, Rupérez, Borroto and Saura-Calixto 1996, Grigelmo-Miguel et al. 1999, Grigelmo-Miguel and Martín-Bellosa 1999, Larrauri 1999, Nawirska and Kwaśniewska 2005). This procedure determines soluble dietary fibre and lignin. Similar to the enzymatic-gravimetric, starch and/or protein is hydrolysed by enzymes. Isolation of soluble dietary fibre in the enzymatically hydrolysed fraction can be achieved by alcohol precipitation or dialysis. Determination of sugars (either by spectrophotometry, gas-liquid chromatography or high-performance liquid chromatography), and uronic acids (colourimetry) can also be performed to obtain more information if desired. The insoluble fraction collected from enzymatic treatment is further hydrolysed by sulfuric acid to obtain acid non-hydrolysable residue quantified as Klason lignin (Englyst et al. 1994, Manas et al. 1994).
9.6 Potential use of bioactive compounds from fruit juice industry waste

The potential use of phenolic compounds and dietary fibre products from fruit juice wastes as novel functional food ingredients is has very high potential for/in the food industry. Over the last few decades, the demand on functional food has increased as consumers are more health-conscious and expect food to deliver health-promoting physiological effects on top of providing nutrients and satiety. The global functional foods market was worth an estimated USD 43.27 billion in 2013. In comparison to the market values of year 2009, this figure has increased by 26.7%, and continues to demonstrate annual growth in excess of the world food industry as a whole (Leatherhead Food Research 2014). Functional food ingredients derived from natural sources are highly sought-after in order to deliver products matching the consumers’ demands on functional foods of natural ingredients. Due to this driving force, bioactive compounds recovered from fruit wastes not only provide a solution to food manufacturers in terms of affordability and availability of the ingredients they are seeking, but also a more sustainable approach of using valuable resources which become more and more limited. Phenolic compounds and dietary fibre recovered from various fruit wastes has been introduced into various types of food as functional additives, such as antioxidative, colouring, antimicrobial agents, as well as texture modifiers.

Kabuki et al. (2000) reported that mango seed kernel ethanol extract exhibits antimicrobial activities against a broad spectrum of bacteria, especially gram-positive. The antimicrobial activity of the mango seed kernel extract was stable against sterilization conditions, freezing conditions, and a wide range of pHs which makes it suitable for use in food processing. Bergamot peel extract exhibited antimicrobial activity against gram-negative bacteria (Mandalari et al. 2007). Fattouch et al. (2008) compared polyphenolic profiles, and antioxidant and antimicrobial activities of pome fruit peels (apple, pear, and quince) and reported that apple and quince peel extracts were effective in inhibiting the growth of *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Bacillus cereus*. Extracts prepared from mangosteen pericarp exhibited strong pH-dependent bacteriostatic and bactericidal effects against *Listeria monocytogenes* and *Staphylococcus aureus* (Palakawong et al. 2013). Casquete et al. (2015) reported the citrus peel extracts (lemon, mandarin, sweet orange) demonstrated antimicrobial activity against a wide range of microorganisms and high pressure treatment did not alter those antimicrobial
activities. Promising antimicrobial effects of raspberry pomace extract against *Escherichia coli*, *Salmonella* sp., *Listeria monocytogenes*, *Enterococcus faecium* has also been reported (Caillet et al. 2012). Pomegranate peel extract showed excellent antioxidant activity against *Staphylococcus aureus* and *Bacillus cereus* and helped prolonging the shelf life of chilled chicken products by 2–3 weeks (Kanatt et al. 2010).

With regards to antioxidant activity, phenolic compounds extracted from mango seed kernel powder was reported to prolong the shelf life of buffalo ghee (Puravankara et al. 2000). Apple wastes’ phenolic extracts were found to be as effective natural antioxidants in stabilizing fish-oil (Sekhon-Loodu et al. 2013) and meat products (Yu et al. 2015). Flavanol oligomers obtained from grape pomace were reported as potent inhibitors of oxidation in emulsions and in frozen fish muscles (Pazos et al. 2005).

In many reports on the use of bioactive compounds from fruit wastes in food products, antioxidant activity is reported as having a synergistic effect with the addition of dietary fibre (Saura-Calixto 2010). As described in the previous Section, when dietary fibre is prepared directly from fruit waste without prior extraction step to remove other bioactive compounds, the resulting dietary fibre products generally contain high amount of other bioactive components associated to the fruit source. Due to this, many reports on waste-derived dietary fibre as an antioxidant carrier can be found in the literature. Fruit-waste-derived dietary fibre products have low-caloric value and offer some functional properties, such as water-holding capacity, swelling capacity, increasing viscosity or gel formation which are essential in formulating certain food products. Addition of such dietary fibres into baked goods has been reported to improve functional properties of the doughs as well as the finished products (Sudha et al. 2007, Vergara-Valencia et al. 2007, Ajila et al. 2008, Min et al. 2010, Sivam et al. 2011, Pečivová et al. 2014, Chareonthaikij et al. 2016). Functionality improvement (e.g. rheological improvement, SDF/IDF and dietary fibre level modifier, shelf-life extension, and fat replacement) after the addition of dietary fibres into other food products such as beverages (Sun-Waterhouse et al. 2010, Sun-Waterhouse et al. 2014), dairy (Sah et al. 2016), fish and meat (Cengiz and Gokoglu 2005, Sánchez-Alonso et al. 2007, Sáyago-
Ayerdi et al. 2009), pasta (Ajila et al. 2010), and ready-to-eat snacks (Kayacier et al. 2014, O’Shea et al. 2014) have also been reported.

Apart from direct food product applications, another promising potential application of bioactive compounds recovered from fruit wastes is in the development of active food packaging. The biological activities of phenolic compounds (particularly antimicrobial and antioxidative activity) and technological properties of dietary fibres (water permeability, viscosity, gelling and network formation) make it feasible to develop food packaging with enhance functionality (Appendini and Hotchkiss, 2002, Lopez-Rubio et al. 2006, Janjarasskul and Krochta 2010, Arcan and Yemenicioğlu 2011, Martinez-Avila et al. 2014, Salgado et al. 2015).

9.7 Conclusion

The global market and production values of fruit juice has increased with the drives of production technology and functional food demands. Consequently fruit juice industry generates a huge quantity of waste. Alternative valorisation of fruit waste needs to be addressed as conventional disposal methods are not the best way to utilise such materials. Fruit solid wastes from juice industry contain high levels of recoverable bioactive compounds associated with human health benefits and can be used as cheap sources for the production of these high-value compounds. Extensive studies on extraction, separation, and characterisation of phenolic compounds and dietary fibres from various fruit wastes have been conducted. Nevertheless, more research is still needed throughout the recovery process, such as applications of ‘green’ extraction approaches, and more powerful separation and characterization techniques, in order to achieve higher yield and quality of bioactive extracts suitable for food applications. In the food industry, the recovered bioactive compounds have tremendously high potential uses in the development of functional foods and active food packaging.
9.8 References


potential of exotic fruit byproducts as a source of food additives. Food Res. Int. 44: 1866-1874.


Da Porto, C., E. Porretto and D.a Decorti. 2013. Comparison of ultrasound-assisted extraction with conventional extraction methods of oil and polyphenols from grape (Vitis vinifera L.) seeds. Ultrason. Sonochem. 20: 1076-1080.


Extracts from mango peel by-product obtained by supercritical CO$_2$ and pressurized solvent processes. LWT-Food Sci. Technol. 62: 131-137.


Laufenberg, G., B. Kunz and M. Nystroem. 2003. Transformation of vegetable waste into value added products:: (A) the upgrading concept; (B) practical implementations. Bioresource Technol. 87: 167-198.


Medina-Meza, I.G. and G.V. Barbosa-Cánovas. 2015. Assisted extraction of bioactive compounds from plum and grape peels by ultrasonics and pulsed electric fields. J. Food Eng. 166: 268-275.


Molero Gómez, A., C. Pereyra López and E. Martinez de la Ossa. 1996. Recovery of grape seed oil by liquid and supercritical carbon dioxide extraction: a comparison with conventional solvent


