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Beneficial Ingredients from Kinnow Peel -Extraction and Uses: A Review

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The peel and pomace part of kinnow which is the major waste generated from the juice industry of kinnow constitutes 30-40% of the fruit weight is a rich source of polyphenols, flavonoids such as naringin, carotenoids, pectin and essential oils. These bioactive compounds have great value in industries like pharmaceuticals, food processing, and biofuel production and have good nutritive value as well. The useful compounds can be extracted using different techniques having high efficiency and can be used for different purpose. The cheap and easy availability of the Kinnow peel, which is otherwise a waste, makes it more useful in the industry as it costs low and has multifunctional uses. The usage of the kinnow peel is environment friendly as it reduces wastage and promotes sustainability.

Introduction

Kinnow mandarin (*Citrus reticulata*) which is a hybrid of two cultivars- 'King' and 'Willow leaf' mandarin is an important fruit among citrus fruits in terms of nutritive value as well as economic value. The fruit is mostly consumed as fresh fruit and juice extraction is equally done. The by -product after the juice extraction or fresh consumption is the peel and pomace. The kinnow peel is a rich source of various bioactive compounds and essential oils among which are polyphenols, carotenoids (lutein), pectin, naringin, antioxidants which have industrial importance. Polyphenols present in the peel such as naringin, limonene and the flavonoids are said to have anti-inflammatory and anti-cancer properties (Tripoli *et al.*, 2007; Xu *et al.*, 2019). It is been suggested that the kinnow peel which constitutes 30-40% proportion of fruit is rich in bioactive compounds and have more polyphenol content as compared to other portion of the fruit (Lim *et al.*, 2007). Thus the important properties of citrus peel have prompted the researchers to explore the scope of this waste product as a valuable component for the processing industries (Babbar *et al.*, 2011; Rafiq *et al.*, 2018). The kinnow peel consists of two different tissues are found namely albedo (inner layer) and flavedo (outer layer).

Albedo mainly constitutes various nutrients viz. minerals, sugars, proteins and fibre (Marin *et al*., 2007). On the other hand flavedo constitutes essential oils which are useful for flavour and fragrance industry (Bejar *et al*., 2012). The essential oils present in the flavedo layer are rich in carotenoids, terpenes and linalool (Mondelaa *et al*., 2005) and it is suggested by various researchers that these bioactive compounds have antimicrobial properties and act as antioxidants (Tepe *et al*., 2006; Jayprakasha *et al*., 2008; Viuda-Martos *et al.,* 2008). Thus the fact that that the peel portion of the fruit have comparatively more phenolics and have high potential for its useful ingredients than the fruit pulp (Deng *et al.,* 2012; Goulas *et al.*, 2012; Jabbar *et al.*, 2015) has made the scope for the study of the constituents, their extraction and use for the industry.

Extraction of useful bioactive compounds

The useful bioactive compounds in kinnow peel such as polyphenols, carotenoids, pectin, naringin, limonene and essential oil is done at industry level using different techniques and a variety of researchers have experimented on these processes. Thus following is the account of the extraction process of these useful compounds and there further uses.

Polyphenols

Polyphenols are natural antioxidants which are found in fruits and vegetables and are proven beneficial for the human body due to their properties of free radical scavenging and chelation of pro oxidant ions in the body (Wojdylo *et al*., 2007; Osman *et al.,* 2007). Various studies suggest that there is decrease in cardiovascular disease, cancer and other degenerative diseases with increased intake of fruits and vegetables due to the antioxidant activities of phenolic compounds present in them (Huang *et al.*, 2011; Sesso *et al.*, 2012)

The extraction of polyphenols is done by solvent extraction methods, the organic solvents which are used for the extraction include ethanol, methanol, ethyl acetate and acetone (Ross *et al*., 2007). The methods for extraction include conventional as well as non-conventional methods. The conventional extraction method which is commonly used is maceration technique (Belova *et al*., 2009).The other conventional methods include stirring, blending, shaking, ultrasonic extraction and rotary extraction (Jeong *et al.*, 2004; Peleg *et al.*, 1991; Wang *et al.*, 2008; Xu *et al*., 2008; Y.Q. *et al*., 2008; Ma *et al*., 2009). The conventional methods have their own limitations such as low efficiency, lower yield and associated health hazards (Jadhav *et al*., 2009; Adje *et al.,* 2010). The non-conventional extraction technique is Ultrasound-Assisted extraction (UAE) which involves the mixing of sample with organic solvents and placing it in ultrasounds for a specific time and temperature, the sound waves generate cavitation and rupture the cell wall of the sample resulting in extraction of phenolic compounds to the solvent medium (Da Porto *et al.*,2009). It is been found by various researchers that the UAE process takes less than an hour and yield 6-35% more than the traditional methods which take more time which is 12 hours or more (Jabbar *et al.*, 2015; Vilkhu *et al.*,2008). Other than UAE there are several other non-conventional and more efficient techniques which are developed due to increasing demand in this sector. These techniques include Microwave-Assisted extraction (MAE), supercritical fluid extraction and accelerated solvent extraction (Barbero *et al.*, 2008). MAE include heating of the solvent having the solid sample by the use of microwaves and dissecting the required compounds from the solvent and this technique is more preferred due to less time consumption and energy saving (Spignoet *et al.,* 2009). Therefore the non-conventional methods of polyphenol extraction are preferred over the conventional methods due to time saving and more efficiency.

Microwave assisted extraction (MAE) has also been performed for the extraction of polyphenols from Kinnow peels (Hayat *et al.*, 2009) and the results have been compared with other conventional techniques such as ultrasonic extraction and rotary extraction. The extraction conditions which were found to be optimum are- 152W microwave power, 49 seconds extraction time, 16 liquid to solid ratio and 66% methanol (solvent) concentration. The phenolic acids extracted were gallic acid, p-hydroxybenzoicacid, vanillic acid, p-coumaric acid, ferulic acid. These phenolics were tested for their antioxidant activity and compared with other conventional techniques like ultrasonic extraction and rotary extraction and it concluded that MAE is the most effective technique among them due to its high efficiency and less time consumption. However, the optimal conditions mentioned above should be applied and the increase in the time or heat resulted in the degeneration of the phenolic acids.

Ultrasound-assisted extraction (UAE) and maceration techniques using different solvents for extraction of polyphenols from citrus peels have been used for comparison and quantification of antimicrobial and antioxidant activity of these phenolics (Muhammad *et al.,* 2016). The antioxidant properties were estimated using different assays such as ferric reducing antioxidant power (FRAP), 2,2-diphenyl-1-picrylhydrazyl (DPPH) and superoxide radical scavenging. It has been found during the study that UAE is more efficient as compared to maceration technique and maximum polyphenol yield (32.48mg gallic acid/ g solvent) was obtained in UAE technique using 80% methanol as solvent and the least yield (8.64mg gallic acid/ g solvent) was obtained using maceration technique using 80% ethyl acetate as solvent. The antioxidant activity was also found to be higher when the solvent used was 80% methanol as compared to other solvents.

These techniques are found to be very effective for the extraction of polyphenols and the antioxidant activity of these extracted polyphenols can be quantified. The antioxidant properties can be identified using FARP, DPPH and superoxide radical scavenging assays, however, DPPH assay is found to be more effective and easy to use (Bendini *et al.*,2006; Perez *et al.*,2009).

Pectin

Pectin extracted from the peel has a varied applicability in the food industry globally due to its use in the manufacturing of various food products like jams, jellies, preservatives (Lim *et al.,* 2012). The pectin due to its high content has become highly valued in the food industry as soluble dietary fibre (Sharma *et al.*, 2006). The extraction of pectin is multi step process and mostly the acid extraction process is used where sample solution is acidified using a strong acid, then the mixture is incubated in a shaker and precipitated pectin is obtained by when ethanol (96%) is added to the filtrate obtained in the shaker (Sharma *et al.*,2013).

Implication of water acidified with nitric acid has been done for the extraction of pectin from Kinnow peel and pomace (Sharma *et al.*, 2013). Varied conditions- temperature (40-80 degree Celsius), pH (1.25-2.25) and time (10-90min) were used for the purpose of extraction process. The results indicated that pectin content is comparatively more in peel than pomace and yield of pectin increased with increasing the temperature till 60 degrees and then reduce on further increasing the temperature and also the yield decrease if pH is above 2.0. The study illustrated that highest yield of pectin (16% in peel and 6.2% in pomace) was found at 60 degree Celsius temperature, 1.75 pH and 70 min extraction time.

Carotenoid

The bioactive compounds can be further classified as – Carotenes and Xanthophyll (Lutein). The carotenoids extracted from the peel act as natural colouring agent for food industries due to natural origin, no toxicity and natural pigments which are useful in the food industry (K.A *et al.,* 1996; M.J. *et al.,* 2001). The extraction of the carotenes can also be done using solvent extraction but it is not preferred as the pigment becomes prone to oxidation after extraction, therefore the enzymatic extraction of the carotenoids is preferred. The enzymatic preparations for the extraction process which include CM Case enzyme are preferred as it increases pigment yield by disintegrating the plant tissues (R.C. *et al.,* 2013). The enzymes break the cell wall and the structure to expose the carotenoids for extraction.

The xanthophyll which includes lutein can be extracted using the conventional solvent extraction method such as ultrasound-assisted extraction (UAE) (Saini *et al.,* 2020). These cannot be synthesised in human body, thus need to be taken in the diet. Lutein is responsible for the change of the colour of fruit (yellow to orange). Lutein is also very beneficial for the human body as it prevents cancer; age related blindness and cardiovascular disease and this pose a very useful carotenoid (Fernandez *et al.,* 2010; Lin *et al.,* 2015; Del *et al.,* 2007).

Extraction of carotenoids from Kinnow peels using the cellulolytic enzyme and improving the stability of extracted carotenoids (Nadeem *et al.,* 2018). Highest yield of carotenoid (8.60mg/100g peel) was obtained by the treatment which included both CMCase and pectinase (250IU/100g peel). Also, the stability of the extracted carotenoid pigment was more in at 30 degree Celsius temperature in dark as compared to light and the stability decreases with increase in temperature, therefore freeze drying of the pigment results in more stability. The lutein was extracted using UAE method with methanol solution as extractant and the optimised parameters were- 43.14 degree Celsius temperature, 6.16mL/g solvent/ solid ratio, 33.71 min time duration (Saini *et al.,* 2020). These parameters resulted in highest yield of lutein (29.70μg/g). The yield of the extract is determined using HPLC (High Pressure Liquid Chromatography) technique.

Naringin

Naringin is a flavonoid found in the peel of kinnow especially in immature fruits, it is basically responsible for the bitterness of kinnow peel along with other compounds like limonene but it has important use in pharmaceutical industry and food industry. Naringin is used in pharmaceutical industry as it shows antioxidant properties and anti-cancer activity. It is beneficial for the human body as it is effective in reducing cholesterol levels and protects against carcinogenic compounds and acts against the toxins during chemotherapy (Gorinstein *et al.,* 2006; Jiang *et al.,* 2006). Different methods for recovery of naringin from peels viz- solvent extraction have been used by researchers (Bhatlu *et al.,* 2016 Seshadri *et al.,* 1962; Davis, 1966), supercritical carbon dioxide extraction (Yu *et al.,* 2007), precipitation method (Crandall *et al.,* 1976; Tripodo *et al.,* 2007), adsorption method (Calvarano *et al.,*1996; Jiang *et al.,* 2006). The naringin content in the immature fruits is comparatively more and therefore the pre-harvest dropped kinnow can be utilised for the extraction process and economically beneficial to the growers. Removal of bitterness from the Kinnow peel and pomace by extracting naringin and limonene has been studied (Singla *et al.,* 2020). Solventogenesis method with acetone gave maximum yield of naringin and limonene from kinnow peel and pomace (8.955, 2.122 mg/g from kinnow pomace and 9.971, 3.838 mg/g from pulp, respectively). This method utilises the by-product and gives a sustainable solution.

Kinnow suffers from pre harvest drop and the dropped fruits are a potential source of extraction of naringin and pectin (Bhatlu *et al.,* 2016). The immature kinnow pulp was boiled and the extract was passed through indigenous raisin Indion PA 800 and naringin was adsorbed in it. The adsorbed naringin was desorbed using ethanol, the solution was further filtered and naringin was obtained by evaporating the filtrate. The naringin recovered was 52% with a final purity of 91-93%.

A study conducted by Puri *et al.,* 2010 on the molecular characterization of naringin extracted from kinnow peel waste suggested that rhamnose (an important compound for the food industry) can be synthesized from naringin by using α-l-rhamnosidase. These studies mentioned above suggest the importance of naringin in the various industries and a solution for the utilization of kinnow pulp residue.

Essential oils

A number of researches suggest different techniques for the extraction of essential oils from kinnow peel. Javed *et al.,* (2014) extracted essential oils from fresh kinnow peel using hydro-distillation method procuring yield of 0.33% and required time of 210 min. Similarly, Kamal *et al.,* (2011) performed hydro-distillation method for extracting essential oils and results indicated the range of content of essential oils for fresh peels (0.30-0.50g/100g), ambient peels (0.24-1.07g/100g) and 0.20-0.40g/100g for oven dried peels.

Various methods for the extraction of oil from kinnow peel which include- oil extraction from shredded peel by hydrodistillation, oil extracted from shredded peel by centrifugation, oil extracted from fresh peel by hydrodistillation, oil extracted from mechanical dried peel powder by hydrodistillation, oil extracted from solar dried peel powder by hydrodistillation (Sharma *et al.,* 2019). The highest oil content obtained is from mechanical dried peel powder by hydrodistillation, also the essential oil content obtained was found to be more from peel powder as compared to fresh peel which is apparently due to the rupture of oil glands during powder making process. It also suggested that physical method like centrifugation resulted in less time consumption comparatively and high redness value.

Effect of drying techniques on useful bioactive compounds

The drying of kinnow peel can greatly influence the content and usefulness of the bioactive compounds such as polyphenols, carotenoids, flavonoids, pectin, etc. and therefore it is important to choose the correct technique. The drying techniques which can be useful for the drying of the kinnow peel are vacuum drying, freeze drying, dehydration, etc. Various research work is done to explore the drying techniques in various other fruits and vegetables like okra (Huang *et al.,* 2015), beetroot (Hamid *et al.,* 2018), quince (Izli and Polat, 2018), tropical fruits (Morais *et. al.,* 2015), potato (Eren and Kaymak-Ertekin, 2007), bittergourd (Mudgal and Pandey, 2009), apple pomace cake (Shalini *et al.,* 2009).

Different drying methods have a varying degree of effect on the quantity and quality of bioactive compounds in Kinnow (Rafiq *et al.*, 2019). There is significant decrease in the moisture, phenolic content, flavonoids, colour and antioxidant activity of the dried sample as compared to the fresh sample regardless of the method used for drying which in this case were- Tray drying, vacuum drying and freeze drying. However, it is suggested by the results that freeze drying method was found to retain more moisture, phenolics, flavonoid content and colour (carotenoids) when compared to the other drying techniques.

Use of kinnow peel for candy preparation

Kinnow peel can be used for the preparation of candy using sugar syrup and the potential waste can be utilised into edible candy form. Kinnow peel candy prepared by first drying the Kinnow peel, cut to shape and proper thickness and then cooked with different concentration of sugar syrup at different temperature for different time- I. 50 degree Celsius for 15 min with 40 Brix sugar syrup concentration; II. 60 degree Celsius for 20 min with 50 Brix sugar syrup concentration; III. 70 degree Celsius for 25 min with 60 Brix sugar syrup concentration (Bhatlu *et al.*, 2014) prepared candy using different sugar syrup concentrations. All the samples prepared were optimum for consumption but the third treatment gave more colour (chocolate) and good taste comparatively.

Use of kinnow peel for biofuel production

Kinnow peel is a potential source of energy as it can be used as a bio fuel and a source of carbon nano fibres (Mahmood and Hussain, 2013). The kinnow peel was gasified using cobalt oxide nano catalyst which resulted in the production of following-

60% liquid extract which can be used for production of ethyl ester (Biodiesel) upon esterification.

28% fuel gases which include ethane, propane, and methane, methanol which can be used for production of biodiesel, bio methanol, and hydro carbon fuel.

10-12% charcoal which can be used for production of carbon nano fibres.

Production of microalgae which further facilitates the production of biodiesel has been tested to great success (Chauhan and Amit, 2019). The kinnow peel along with wastewaters like dairy waste water, sewage water was used for the biomass production of algae using three different mediums (KEM, KEMS, KEMD). It was observed that highest bioalgal mass was produced in KEMS medium. The study suggests that using kinnow peel can be an economically viable option for the biofuel production. This is the indirect use of kinnow peel for the biofuel production.

In conclusion kinnow is an important fruit crop with multipurpose utility. Production technology of Kinnow mandarin is basically aimed at increasing the per unit area production but also various value added products and bioactive compounds can be synthesized which can boos the pharmaceutical industries also and generate a sizable amount of revenue.

The following points can be concluded from above review of work of various researchers:

- Kinnow peel and pomace which constitutes 30-40% of the fruit weight which is otherwise a waste product can be utilised for various uses.
- Kinnow peel consists of various bioactive compounds such as polyphenols, flavonoids, pectin, carotenoid which have a high industrial value and nutritive value.
- Some of the bioactive compounds found in kinnow peel like lutein are beneficial

compounds for human body and are not synthesised in the body naturally, thus increasing their importance.

The bioactive compounds need to be extracted using different methods and processes for optimum usage.

The usage of kinnow peel varies from widely and can be used in different industries for different work viz. from being used for candy preparation to being used in biofuel manufacturing industries.

The use of kinnow peel not only reduces the wastage but also promotes sustainable growth and bring economic value to the producers.

Thus kinnow peel is a useful product which is mostly wasted and need to be taken into use more often due to its high nutritive content and wide usage in different sectors.

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