



Mango pectin quality as influenced by cultivar, ripeness, peel particle size, blanching, drying, and irradiation



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ABSTRACT

Industrial recovery and application of valuable mango (*Mangifera indica* L.) peel constituents, such as dietary fiber and pectins, require the conversion of the yet under-utilized and highly perishable by-product into a stable commodity. Focusing on efficient pectin recovery, the impact of different cultivars and ripeness degrees as well as various technological procedures on pectin quality by affecting pectin yield, molecular size distribution of pectic polymers, galacturonic acid content, degree of esterification, and content of interfering substances was analyzed. Cultivar and ripeness degree revealed a significant effect on pectin quality. Preservation processes, i.e. oven drying and lyophilization each with and without previous blanching of integral fruits as well as gamma irradiation, notably influenced the quality of the obtained pectin. Blanching prior to drying reduced arabinogalactan and ash impurities, whereas galacturonic acid contents were increased. Most importantly, grinding of dried mango peels to obtain a particle size of ca. 42 μm (d_{43}) significantly enhanced both extraction yield (+70%) and galacturonic acid content (+20%) without increasing the contents of the above mentioned impurities as compared to a peel particle size of ≥ 10 mm. Mango pectin produced from such peel powders with a small particle size (≤ 120 μm) improved breaking and sugar binding capacities as well as gelling units (up to 5476 GU). The production of mango peel pectin and its applications were favored by implementing the proposed procedures into the valorization cascade of mango peels.

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1. Introduction

Due to its savory taste and high nutritive value (Tharanathan, Yashoda, & Prabha, 2006), worldwide mango (*Mangifera indica* L.) production has continuously increased to 46.7 million mt in 2012 (FAOSTAT, 2014). Besides their sale on fresh markets, value added mango products such as juice, canned pulp, and chutneys are produced at industrial scale. The accruing by-products, i.e. the stones and peels, often generate a major disposal problem for producers. While the fat of the stones can be recovered and utilized as cocoa butter equivalent up to a maximum of 5% in certain EU member states (Directive 2000/36/EC), a valorization process for

the peels is still urgently needed. The recovery of valuable phytochemicals, such as flavonol glycosides, monoterpenes, alkylresorcinols, and, most importantly, mango pectin, has been proposed previously (Neidhart, Sirisakulwat, Nagel, Sruamsiri, & Carle, 2009; Schieber, Stintzing, & Carle, 2001; Sirisakulwat, Nagel, Sruamsiri, Carle, & Neidhart, 2008). The high content in such valuable bioactives and techno-functional compounds as well as their excellent digestibility has been shown in numerous studies (Engels et al., 2009; Geerkens et al., 2013; Geerkens, Matejka, Carle, & Schweiggert, 2015; Larrauri, Rupérez, Borroto, & Saura-Calixto, 1996; Schieber et al., 2001), thus making mango peels a promising target for commercial valorization (Nagel, Neidhart, et al., 2014; Panouillé, Ralet, Bonnin, & Thibault, 2009). However, after producing the primary mango product, e.g., juice or puree, the wet peels are highly perishable and prone to microbial spoilage and endogenic enzymatic degradation reactions. In particular,

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endo- (EC 3.2.1.15) and exo-polygalacturonases (EC 3.2.1.67) as well as pectin methyl esterases (EC 3.1.1.11) represent a serious obstacle for pectin recovery, unless these deteriorative enzymes are inactivated (Ali, Chin, & Lazan, 2004). Although Sirisakulwat, Srumsiri, Carle, and Neidhart (2010) reported only insignificant degradation of the mango peel pectin within 5 h after peeling, middle- and long-term storage of the peels is impeded due to enzymatic decay and microbial spoilage. Hence, post-processing treatments preserving the peels and retaining their valuable constituents were previously recommended (Sirisakulwat et al., 2008). In the EU, the galacturonic acid content of food grade pectins shall be not less than 65% according to EU regulation No 231/2012. While pectins obtained from citrus peel and apple pomace commonly meet this requirement, lower galacturonic acid levels are important quality defects of mango “pectin”. In contrast to food use, there are no regulations for feed use (Regulation 68/2013/EU). However, the application of modified technological procedures has previously been shown to afford galacturonic acid contents greater than 65% (Nagel, Neidhart, et al., 2014).

The first objective of the present study was the stabilization of wet peels by blanching the integral fruits and subsequent drying of the manually obtained peels. By these means, inactivation of pectin-degrading enzymes and prevention of microbial decay should be achieved. Furthermore, the influence of peel particle size reduction for pectin extraction on pectin quality and yield should be analyzed. In addition to blanching, drying, and particle size reduction, the effect of gamma irradiation on mango peel pectin was analyzed, since India is obliged to irradiate fresh mango fruits destined for the US market in order to extend their shelf-life and improve their phytosanitary status (Allothman, Bhat, & Karim, 2009). Moreover, gamma irradiation may exert potential effects on chemical and physical product properties (Chung & Liu, 2009). Beyond investigating the influence of technological procedures, pectin quality and yield from peels of three monoembryonic and a polyembryonic cultivar was compared. Peels from two of these cultivars were obtained in unripe and ripe condition in order to elucidate the effect of fruit ripeness on pectin quality and yield. The chemical and techno-functional properties of all pectins obtained were characterized in detail. Carbohydrate composition including contents of galacturonic and glucuronic acid and seven neutral sugars was analyzed. Furthermore, the degree of esterification (DE), molecular weight distribution, and gelling properties were analyzed. Besides pectin, the total dietary fiber content of the peels was examined, since mango peels were previously shown to contain valuable amounts of total dietary fiber (Ajila & Prasada Rao, 2013; Nagel, Neidhart, et al., 2014).

2. Materials and methods

2.1. Raw material and chemicals

Mimicking industrial pectin extraction by processing fruits of different ripeness degrees, mangoes (*M. indica* L.) of the cultivars Tommy Atkins, Kent, Palmer, and Nam Dokmai were purchased from a local market in Stuttgart, Germany having different maturity, and stored at 13 °C until processing. Peels of cv. Kaew were obtained from Chiang Mai (Thailand), while peels of cv. Totapuri were from Uttar Pradesh (India) and stored in sealed vacuum bags at the University of Hohenheim until used.

The neutral sugar reference standards L-(+)-arabinose, L-(-)-fucose, D-(+)-glucose, D-(+)-galactose, D-(+)-mannose, L-(+)-rhamnose, and D-(+)-xylose, and the uronic acids D-(+)-galacturonic acid and D-(+)-glucuronic acid were purchased from Sigma–Aldrich (Steinheim, Germany). The dietary fiber test kit

Bioquant[®] was from Merck (Darmstadt, Germany). 2-(*N*-morpholino)ethanesulfonic acid (MES) was from VWR International (Darmstadt, Germany), and 2-amino-2-hydroxymethyl-propane-1,3-diol (TRIS) was obtained from Pharmacia Biotech AG (Uppsala, Sweden). Sodium hydroxide solution (50%, w/w) was from J.T. Baker (Avantor Performance Materials, Griesheim, Germany). All other reagents or solvents (analytical or HPLC grade) were purchased from Sigma–Aldrich (Steinheim, Germany) and VWR International (Darmstadt, Germany). Ultrapure water was used throughout. Anhydrous methanolic 2 M hydrochloric acid (HCl) was prepared as described previously (Nagel, Sirisakulwat, Carle, & Neidhart, 2014).

2.2. Mango processing

The ripening index (RPI) of mango fruits was analyzed according to Vásquez-Cañedo, Heller, Neidhart, & Carle (2006). After manual peeling, convective oven drying of peels was carried out for 8 h at 60 °C using a UT 6120 drying cabinet (Hanau, Germany) with and without previous steam blanching of integral fruits for 3 min at 100 °C. Lyophilization of mango peels for 90 h was performed after grinding with liquid nitrogen with and without the above mentioned steam blanching prior to peeling using a Lyovac GT 4 (Oerlikon Leybold Vacuum, Cologne, Germany). Gamma irradiation of mango peels of cv. Totapuri was accomplished with ⁶⁰Co for 3 h, reaching a final dose of 10 kGy as detailed recently (Geerkens, Matejka, et al., 2015).

The dried peels of all cultivars were milled and sieved with a ZM 1 grinder (Retsch, Haan, Germany) equipped with a 0.25 mm ring sieve. Regarding peels from cv. Kaew, greater particle sizes were obtained by sieving the milled peels with a 0.5 mm ring sieve and manual cutting (≥ 10 mm) of the integral peels with a stainless steel knife, respectively. The Sauter mean diameter (d_{43}) of dried mango peel powders was analyzed using a Mastersizer 2000 (Malvern Instruments, Worcestershire, UK).

2.3. Pectin extraction

Hot-acid pectin extraction was performed with 20 g dried and ground or cut peels and 380 g water under continuous stirring at boiling temperature. The slurry obtained was cooled to room temperature in an ice bath, adjusted to pH 1.5 with aqueous sulfuric acid (2 N), and heated at 90 °C for 2.5 h followed by re-cooling to room temperature. The solution was filtered and pressed manually using a nylon cloth. The solid residue retained by the cloth was washed with 200 mL water and pressed. The combined filtrates were added to 3 L of 2-propanol to precipitate alcohol insoluble solids (AIS). The AIS were filtered and pressed using the nylon cloth, washed with 2-propanol, and pressed again. Finally, the AIS were dried at 60 °C for 14 h using a convective oven dryer, milled, and sieved with a ZM 1 grinder (Retsch, Haan, Germany) equipped with a 0.25 mm ring sieve. Pectin extraction was conducted in duplicate for each cultivar and ripeness degree, respectively, according to Geerkens, Miller-Rostek, et al. (2015).

2.4. Quantitation and characterization of dietary fiber

For the determination of total (TDF), soluble (SDF), and insoluble dietary fiber (IDF), the enzymatic-gravimetric AOAC Official Methods 985.29 and 991.43 (1997) were conducted in duplicate using the MES/TRIS buffer solution.

The swelling (SC), water holding (WHC), and oil holding capacities (OHC) were analyzed in duplicate according to Nagel, Neidhart, et al. (2014).

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