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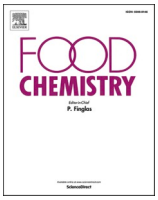

Influence of ripening stage on the microwave-assisted pectin extraction from banana peels: A feasibility study targeting both the Homogalacturonan and Rhamnogalacturonan-I region

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ABSTRACT

This work investigated a sustainable and efficient approach of pectin extraction for banana peel waste valorisation and studied the influence of banana ripening stages (RS at 2,5 and 7). Although pectin content in banana peel raw material decreased during ripening, pectin extraction was favoured. The highest alcohol-insoluble solids (AIS) yield (12.5%) was achieved at 70 °C, 15 mins from RS 7 peel. All extracts were homogalacturan-rich with some rhamnogalacturonan-I content (showing HGA/RG-I ratio > 2) with varied degree of methylation (DM). The highest HGA content (837.2 mg/g AIS) and HGA/RG-I ratio (9.9) were achieved at 110 °C, 0 mins from RS 7, suggesting its promising application as gelling agent. The highest RG-I content (111.1 mg/g AIS) were obtained at 110 °C, 5 mins from RS 7, which was comparable with the pectin with reported prebiotic ability isolated from the literature, suggesting its potential application in novel products.

1. Introduction

Banana is a general term for a group of species or hybrids in the genus *Musa* of the family *Musaceae*. It is one of the most consumed tropical fruits in the world. The global production of banana reached 113.3 millions of tonnes per annum in 2020 (FAOSTAT, 2021). Banana is a kind of climacteric fruit that continues to ripen after harvest with increased ethylene production and a rise in cellular respiration, through which the colour, texture, flavour, nutritional value and aroma of the banana dramatically change (Alexander & Grierson, 2002). As shown in Fig. 1, the ripening of banana can be divided into 7 stages based on a colour change from green to yellow. Generally, banana is green when harvested, transported over long-distance, and then ripened at the destination using controlled ethylene gas before being marketed at stage 5 or 6 (Soltani, Alimardani, & Omid, 2010). Banana ripening is the work of various enzymes and is influenced by the change in turgor pressure, the degradation of starch and cell wall polysaccharides (cellulose, hemicellulose and pectin) and the dehydration and/or loss of dry matter (Tucker & Grierson, 2013).

With the development of the food processing industry, bananas have been used to make chips, flours, jams, spirits distilled from wine of beer, as well as processed banana products (Emaga, Andrianaivo, Wathelet, Tchango, & Paquot, 2007), and this generates a growing amount of banana waste. Banana peel accounts for approx. 30–40% of the total weight of the fruit, and the waste is usually discarded into landfill or as low-value animal feed (Gomes, Vieira, Barbosa, & Pinheiro, 2022). Due to the high water content and large amount of nitrogen and phosphorus in banana peel, it is highly susceptible to microbial degradation and therefore can cause environmental problems if not disposed effectively (Oliveira et al., 2016). On the other hand, recent research has reported potential health benefits of banana peel, such as antioxidant, antibacterial and antibiotic properties, which attribute to its high dietary fibre contents and various phenolic compounds (Zaini et al., 2022). One of the solutions to the adverse environmental impact and economic losses associated with banana peel waste is to achieve valorisation by extracting high value co-products from them. A valuable co-product that can be obtained from banana peel waste is pectin.

Pectins are complex heteropolysaccharides that can be found in the primary cell wall and middle lamella of terrestrial plants, where they functions as a gel between lignocellulosic structures assisting with cell

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adhesion and softening cell walls during cell ripening through enzymatic hydrolysis (Silva & Rao, 2006). The structures of pectin can be mainly divided into two domains: 1). Homoglacturonan (HGA), a linear homopolymer of α-1,4-linked-d-galacturonic acid (GalA.) methylesterified and acetylated to various degrees and 2). Rhamnogalacturonan-I (RG-I), a galacturonan main chain interrupted by the regularly repeating insertion of L-rhamnose residues to form a S-D-GalpA-1,2-S-L-Rhap-1.4 backbone and with various D-galactose and L-arabinose side chains connected to the L-rhamnose residues (Mao et al., 2019). Banana peel pectin is specifically rich in HGA region (40.2–71.8%) and also contains some RG-I region (2.6–10.9%) (Emaga, Ronkart, Robert, Wathelet, & Paquot, 2008).

The structural features of pectin define its applications. HGA-rich pectins are most widely used as hydrocolloids due to their ability to trap or bind water to form gels at low concentration, although the suitability for application is governed by the structural features, including molecular weight, structure linearity, the degree of methylation (DM) and acetylation (DA) etc. (Rasidek et al., 2021). RG-I-rich pectins are not suitable for this application as they do not gel; however, attributing to the presence of the side chains, emerging research has demonstrated their potential in pharmaceutical applications such as prebiotics (Gómez, Gullón, Yáñez, Schols, & Alonso, 2016).

There is arising interest in using microwave-assisted extraction (MAE) in pectin recovery due to its potential ability to achieve enhanced extract yields and quality in a shorter time, and reduced energy and solvent consumption, which are attributed to the unique volumetric and selective microwave heating mechanisms (Mao, Robinson, & Binner, 2021). This is because microwaves can penetrate the whole volume of a material instantly and simultaneously, resulting in a uniform heating throughout the bulk (Mao, Robinson, & Binner, 2023). It can also selectively heat different components in a heterogeneous system at different rates determined by the dielectric properties, which leads to a range of microwave-enhanced mass transfer effects (Mao et al., 2023).

Several studies of MAE for banana peel pectin extraction are presented in the literature, investigating parameters such as the influence of microwave power, extraction temperature and time (Aklilu, 2021;Oliveira et al., 2016; Swamy & Muthukumarappan, 2017). However, the effect of the ripening stage of banana peel on pectin extraction during MAE has not been investigated. This is important because the literature clearly reports the significant alteration of the chemical composition of banana peel cell wall during ripening, including the cleavage of pectin linkages to other cell wall polysaccharides and depolymerisation of the galacturonate backbone (Harding et al., 2017), resulting in an increase in pectin solubility, a decrease in DM, and loss of neutral sugar side chains (Emaga et al., 2007). Most investigators used fully ripe peels (ripening stage 6 or above) because that is the ripening stage that the highest pectin yield is reported in the literature (Oliveira et al., 2016; Swamy & Muthukumarappan, 2017). Moreover, those studies implemented multi-factor experimental design (such as Response Surface Methodology, where multiple variables are changed simultaneously by performing a pre-defined and limited number of experiments) allowing faster determination of optimum extraction conditions with reduced experimental runs. However, this approach can lead to limited optimisation of data and understanding of the role of each experimental variable in reality (Mao et al., 2023). There lacks a systematic experimental approach (such as single-factor controlled experiments) to allow a more thorough investigation of the role of each experimental variable on the pectin extraction yield and composition.

Additionally, pectin composition and extractability can also be influenced by different isolation techniques (Mao et al., 2019). Banana peel pectins are often extracted using acids, which target the pectin HGA region (Castillo-Israel et al., 2015; Lepilova, Aleeva, Koksharov, & Lepilova, 2023; Rivadeneira et al., 2020). However, this method is not suitable for the recovery of RG-I region as the acids attack the side chains, and therefore limits the application of the banana peel pectin recovered. Those acidic extractions (often conducted at pH 1–3) also result in large quantities of acidic waste, posing potential issues in process operation and waste disposal. In the meantime, to the best of authors' knowledge, banana peel pectin extraction at neutral pH has not be reported and the potential to extract unconventional pectin has not been assessed.

To address the abovementioned knowledge gaps in the current state-of-art in banana peel valorisation through pectin extraction, the aim of this study is to investigate how the stages of banana ripening influence pectin extraction, with a view to informing process and raw material selection for tailored pectin-based applications. Water was selected as the solvent owing to its cheap, green and sustainable nature, and more importantly its ability to recover both HGA and RG-I region (Mao et al., 2019). The specific objectives are:

1. Comparing the yield, purity and extraction conditions (temperature and time) on banana peel pectin extracted at ripening stages 2, 5 and 7 using microwaves-assisted extraction through single-factor controlled experiments varying only one variable at a time (e.g. banana peel ripening stage, temperature, time) while carefully controlling all other independent factors (e.g. microwave power, reaction vessel, sample volume, stirring speed etc.).
2. Understanding the impacts of banana ripening stage and extraction conditions on the chemical composition and structural features of the pectin isolated through comprehensive polysaccharides characterisations by sugar analysis (Uv-Vis and HPLC), methylation and acetylation determination (^{1}H NMR) and intrinsic viscosity measurement (viscometer).
3. Proposing different routes for banana peel valorisation and applications for banana peel pectin, focusing not only on the HGA region for traditional application as gelling agents but also the RG-I region, hoping to expand the applications of banana peel pectin into novel and value-added realms.

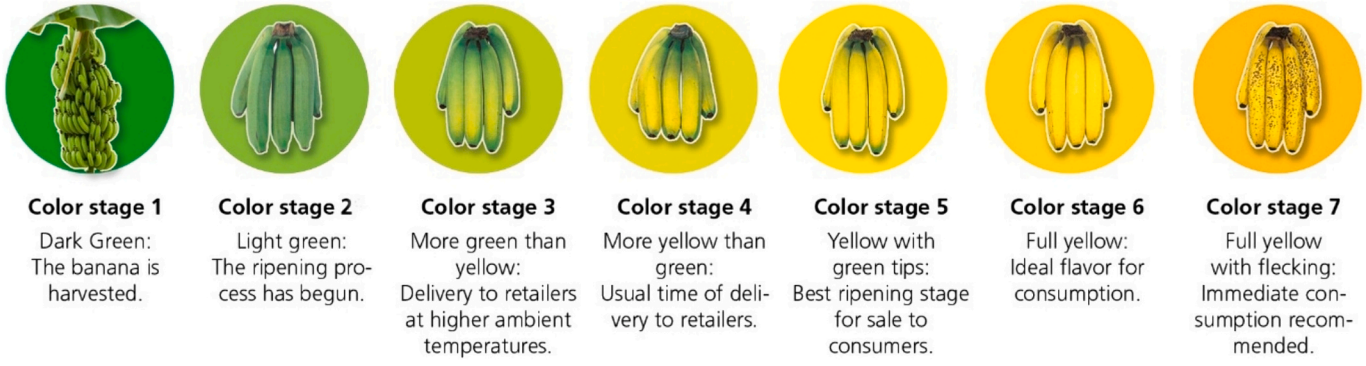

Fig. 1. Stages of banana ripening. (Figure was reproduced from an online source (BioTropicGmbH., 2018)).

2. Material and methodology

2.1. Materials

Banana peels at ripening stage (RS) 2, 5 and 7 were chosen for investigation. Fresh bananas at RS 2 (light green) were bought from a local supermarket (Tesco, Nottingham, UK). Bananas were stored at room temperature until RS 5 (yellow with green tips) and 7 (full yellow with flecking) based on the colour changes according to Fig. 1. Before processing, the bananas were washed and then rinsed with deionised (D.I.) water. The fruit peels were removed from the pulp. The upper stem and bottom black end of the peels were removed, and then the peels were hand sliced into 1 cm squares. To best exclude the influence due to the nature and original chemical compositions difference between different bananas, the sample materials were purchased/prepared in bulk and frozen in batch storage bags. Samples were defrosted overnight in the 4 °C fridge before the day of extraction.

To calculate the sample moisture content, 10 g of banana peels at three RS were dried in the oven at 60 °C until a constant mass was obtained.

2.2. Microwave-assisted extraction (MAE) of banana peel pectins

Microwave-assisted extraction of banana peel pectins was carried out using a Monowave 200 single mode microwave (Anton Parr, St Albans, UK) at 2.45 GHz. Sample solid to liquid (S/L) ratio and total sample volume were the same for all experiments, with 4.5 g of fresh diced banana peels and 15.5 mL of D.I. water. The samples were heated to temperature (50–170 °C) and held at the temperature for the required amount of time (0, 5, 10 and 15 min). The maximum microwave power was 800 W. The samples were then cooled under compressed air to 70 °C before the system was opened. Stirrer speed was set at 800 rpm. After heating, solid banana peel residues were removed by filtration and the resulting liquid was collected for pectin precipitation. An equal volume of isopropanol (IPA) was added to the liquid, and samples were then left to precipitate overnight at 4 °C. The samples were centrifuged for 40 min at 3900 rpm and the supernatant was discarded. The precipitates were called the alcohol-insoluble solids (AIS) and were collected in pellet form, which were freeze-dried using a LyoDry freeze dryer (Mechatech Systems, Bristol, UK).

2.3. Galacturonic acid (GalA.) content analysis

The galacturonic acid (GalA.) content in the raw banana peels and freeze-dried AIS were determined, based on Mao et al. (2019). The HGA regions were firstly decomposed into GalA. monosaccharides by acid hydrolysis using 4 M potassium sulphamate and 2.5 mL of concentrated sulphuric acid and heated to 99 °C for 20 min. A reagent containing 0.15 wt% *m*-hydroxydiphenyl in 0.5 wt% NaOH solution was added into the hydrolysate to allow a pink colour to develop. The galacturonic acid contents were then determined using a UV–Vis spectroscopy (Jenway model 7315, Cole-Parmer Ltd. UK) at a wavelength of 525 nm. Zero absorbance reference was set with deionised water. The standardisation curve was obtained using D-(+)-Galacturonic acid monohydrate (Sigma Aldrich, Dorset, UK) at concentrations from 0 to 97 mg/L and treated the same way as above.

2.4. Neutral monosaccharide content analysis

The raw banana peel and the freeze-dried AIS extract were hydrolysed with concentrated sulphuric acid using the same method as in the GalA. assay. The contents of neutral monosaccharides of the banana peel AIS were determined by a high-performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD) (Dionex, UK) with a CarboPac PA20 column, following the method from Mao et al. (2019). Mixtures of sugar standards (L-rhamnose, L-arabinose, D-galactose, D-glucose and D-xylose (Sigma Aldrich, Dorset, UK)) at various concentrations (1 ~ 20 mg/L) were used as external standards for identification and quantification.

2.5. Mathematical representation and calculation

The extract quantity was determined by yields. The AIS yield represented the percentage of total extract quantity to dry amount of raw banana peel materials, and it was calculated by Eq. (1):

$$AIS\ Yield\ (\%) = \frac{Weight\ of\ freeze-dried\ alcohol-insoluble\ solids\ (AIS)}{Weight\ of\ banana\ peel\ added \times (1 - Moisture\ content)} \times 100\% \quad (1)$$

The extractability of HGA and RG-I regions were determined as the percentage of HGA and RG-I region content in the AIS extracts to the total amount of respective regions presented in the raw material (Kaya, Sousa, Crepeau, Sorensen, & Ralet, 2014).

$$HGA\ extractibility\ (\%) = \frac{HGA\ content\ in\ AIS \times AIS\ Yield\ (\%)}{HGA\ content\ in\ raw\ banana\ peel} \times 100\% \quad (2)$$

$$RG-I\ extractibility\ (\%) = \frac{RG-I\ content\ in\ AIS \times AIS\ Yield\ (\%)}{RG-I\ content\ in\ raw\ banana\ peel} \times 100\% \quad (3)$$

where, the content of HGA and RG-I region were quantified by monosaccharide contents that made up the regions (Alba, Laws, & Kontogiorgos, 2015):

$$\text{HGA content}\ (mg/g) = \text{GalA.–Rha.} \quad (4)$$

$$RG-I\ content\ (mg/g) = 2Rha. + Ara. + Gal. \quad (5)$$

$$Total\ content\ of\ tested\ sugar\ (mg/g) = GalA. + Rha. + Ara. + Gal. + Glu. + Xyl. \quad (6)$$

2.6. Degrees of methylation (DM) and acetylation (DA) analysis

Raw banana peels and freeze-dried AIS extracts were incubated with 1 mL 0.4 M NaOH in D_2O and 0.1 mL of internal standard (TSP, 0.2 mg/mL in D_2O) for 2 h at the room temperature. The solutions were centrifuged, and the supernatants were collected for analysis. The degree of methylation (DM) and acetylation (DA) were determined by ^1H NMR, based on the method from Mao et al. (2020).

DM and DA were calculated as the ratio of the molar percent of methanol or acetic acid to the molar percent of GalA., as in the following equations:

$$DM = \frac{mol\ of\ methanol}{mol\ of\ GalA.} \times 100\% \quad (7)$$

$$DA = \frac{mol\ of\ acetic\ acid}{mol\ of\ GalA.} \times 100\% \quad (8)$$

2.7. Intrinsic viscosity ([η]) analysis

The intrinsic viscosity values of banana peel AIS were measured using a U-tube capillary viscometer (Camlab, UK) at 25 °C. AIS was dissolved in a pH 7 buffer, which was made of 4.595 g/L sodium phosphate dibasic dodecahydrate ($Na_2HPO_4{\cdot}12H_2O$), 1.561 g/L potassium dihydrogen phosphate (KH_2PO_4) and 2.923 g/L sodium chloride (NaCl). The intrinsic viscosity of pectin was determined graphically by extrapolation of Huggins, Kraemer and Solomon-Ciuta plots to zero concentration, following the method from Mao et al. (2020).

2.8. Statistical analysis

To achieve comprehensive and systematic comparison of different extraction conditions (raw material ripening stages, extraction time and temperature), controlled single-factor experiments were performed by changing only one variable at a time while carefully controlling all other independent factors (e.g. microwave power, reaction vessel, sample volume, stirring speed etc.). The AIS yields were calculated based on triplication and the three freeze-dried AIS from each repeat were combined to exclude the difference of each extraction in further analytical characterizations. The results and error bars of various AIS characterizations (GalA., neutral monosaccharides, degree of methylation and acetylation, and intrinsic viscosity) were determined by 4 repeats ((2 freeze-dried samples) × (duplication measurements of each sample by respective techniques), such that the error bars include both the errors from extraction method and characterisation method). All data were expressed as mean values ± standard deviation (SD) ($n = 3$ or 4). Statistical analyses of data were performed using SPSS Statistics (IBM SPSS Statistics, Version 29.0., USA). Data were subjected to a one-way ANOVA with the Duncan method and the probability value of $p < 0.05$ was considered significant.

3. Results and discussion

3.1. Compositional changes in raw banana peels

The changes of chemical compositions of raw banana peels during ripening can be found in Table 1. The moisture contents of raw banana peel decreased slightly from 89.7% to 85.3% wet basis during the ripening. Similar trend was reported for banana ripening by Emaga et al. (2007) and avocado ripening by Liu, Robinson, Madore, Witney, and Arpaia (1999). The decrease of moisture content in banana peel may be due to fruit size expansion and fruit weight increase during ripening (Liu et al., 1999) and the migration of water from peel to pulp due to the difference in osmotic pressure (Adao & Glória, 2005).

The chemical compositions of raw banana peel underwent dramatic changes during ripening. The total tested sugar contents, which were characterised as the sum of all six measured monosaccharides in this study (GalA. + Rha. + Ara. + Gal. + Glu. + Xyl.), firstly decreased from 780.4 to 647.2 mg/g biomass from RS 2 to 5, and then slightly increase to 664.4 mg/g biomass at RS 7. The decrease of total sugar content may indicate pectin degradation as ripening progressed and similar results again were reported by Emaga, Robert, Ronkart, Wathelet, and Paquot (2008). Starch degradation occurred alongside the pectin and formed Glu., which stayed in the cell wall in the form of monosacharride (Sultan & Johari, 2017). It was shown that the amount of Glu. increased from 196.7 to 358.4 mg/g biomass during the ripening.

The most pronounced ripening-associated changes occurred in the pectin composition, which is associated with several pectin-degrading enzymes under very complex working mechanisms and largely dependent on the fruit type and genetics (Uluisik et al., 2016; Wang, Yeats, Uluisik, Rose, & Seymour, 2018; Yang et al., 2017). Firstly, the HGA pectin region (mainly characterised by GalA. content) decreased dramatically from 467.7 to 217.8 mg/g biomass, which was due to the depolymerisation of the galacturonic backbone. Polygalacturonase (PG) is found to be the primary enzyme in vivo, which targets the α1,4-glycosidic bonds between the GalA. residues in HGA (Prasanna, Prabha, & Tharanathan, 2007). Secondly, the RG-I pectin region (characterised by the contents of Rha, Ara. and Gal.) also decreased from 87.7 to 67.4 mg/g biomass from ripening stage 2 to 5; but stayed constant from RS 5 to 7. Rhamnogalacturonase (RGase) is reported as the key working enzyme in RG-I depolymerization, which specifically catalyses the hydrolysis of glycosidic bonds between GalA. and rhamnose units that made up the RG-I backbone (Prasanna et al., 2007). However, experimental results from the present study show increased contents of Rha. from RS 2 to 5, which would not be expected if RGase activity is the predominant factor in the results. This may be due to the hindered activity of RGase by the presence of ester-linked acetyl or methyl groups and/or rhamnose arrangements in the banana RG-I backbone (I. R. Silva, Jers, Meyer, & Mikkelsen, 2016). In the meantime, the contents of Ara. and Gal., which are side chains connecting to Rha., decreased, suggesting the further detachment of side chains from the RG-I backbone and reduced 'hairyness' of RG-I region. From RS 5 to 7, the contents of these three monosaccharides stayed stable, indicating the RG-I region structure stayed stable during the later stage of ripening as opposed to the earlier stage of ripening. The selective hydrolysis of arabinan and galacturan side chains in the RG-I region are due to the presence of arabinase and galactanase enzymes (Prasanna et al., 2007).

3.2. AIS yields

The yields of banana peel AIS extracted comparing ripening stage 2, 5 and 7 are presented in Fig. 2. Overall, banana peels at higher ripening stages yielded not only higher AIS but also at milder extraction conditions (lower temperature and/or shorter times). The highest AIS yields achieved at the tested range of extraction conditions were 6.2% (RS = 2, 110 °C and 10 mins), 9.4% (RS = 5, 110 °C and 10 mins) and 12.5% (RS = 7, 70 °C and 15 mins; or comparatively 11.9% at 90 °C and 10 mins). Despite that the pectin content decreased during ripening as reported in Section 3.1 and Table 1, this AIS result indicates that pectin is more weakly attached to the extracellular matrix as ripening progressed (Brummell, 2006), and therefore becomes more available to extract. The AIS yield achieved in the present study were comparable with those from the literature. For example, Aklilu (2021) was able to recover 5.85% to 16.25% of AIS yield using MAE at temperatures of 60–90 °C and various extraction times (60–100 min) with the optimum condition achieved at 90 °C and 80 mins, though the ripening stage of banana was not mentioned. Rivadeneira et al. (2020) obtained an optimum AIS yield of 14.2% from matured banana peel using MAE with pH 3 HCl solution, 1000 W, 195 °C and 1 mins. However, different literature reported different trends of AIS yields through banana ripening as found in this study, which may be due to the different extraction techniques and conditions used. For example, Emaga, Robert, et al. (2008) reported the increased banana peel AIS yields from 10.3% to 21.7% from ripening

Table 1
Moisture and sugar compositions in raw banana peels at ripening stage (RS) 2, 5 and 7.

	Moisture content	GalA.	Rha.	Ara.	Gal.	Glu.	Xyl.	Total tested sugar	HGA region	RG-I region
	% wet basis	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass	mg/g biomass
RS = 2	89.7 ± 0.0^{Aa}	478.3 ± 2.0^{Ba}	10.6 ± 0.7^{Ca}	29.0 ± 0.3^{Db}	37.5 ± 0.4^{Ec}	196.7 ± 0.5^{Fa}	28.4 ± 0.4^{Gb}	780.5 ± 4.3^{Hc}	467.7 ± 2.7^{Ic}	87.7 ± 2.1^{Jb}
RS = 5	88.5 ± 0.1^{Ab}	320.4 ± 1.8^{Bb}	13.0 ± 0.3^{Cb}	24.0 ± 0.6^{Da}	17.4 ± 0.4^{Eb}	250.5 ± 4.5^{Fb}	21.9 ± 0.4^{Ga}	647.2 ± 3.2^{Ha}	307.4 ± 2.1^{Ib}	67.4 ± 1.6^{Ja}
RS = 7	85.3 ± 0.7^{Ac}	231.1 ± 1.6^{Bc}	13.3 ± 0.6^{Cb}	24.2 ± 0.7^{Da}	15.9 ± 0.4^{Ea}	358.4 ± 1.8^{Fc}	21.5 ± 0.6^{Ga}	664.4 ± 2.7^{Hb}	217.8 ± 1.9^{Ia}	66.7 ± 2.3^{Ja}

Different letters in the same data column indicate the significant difference ($p < 0.05$).

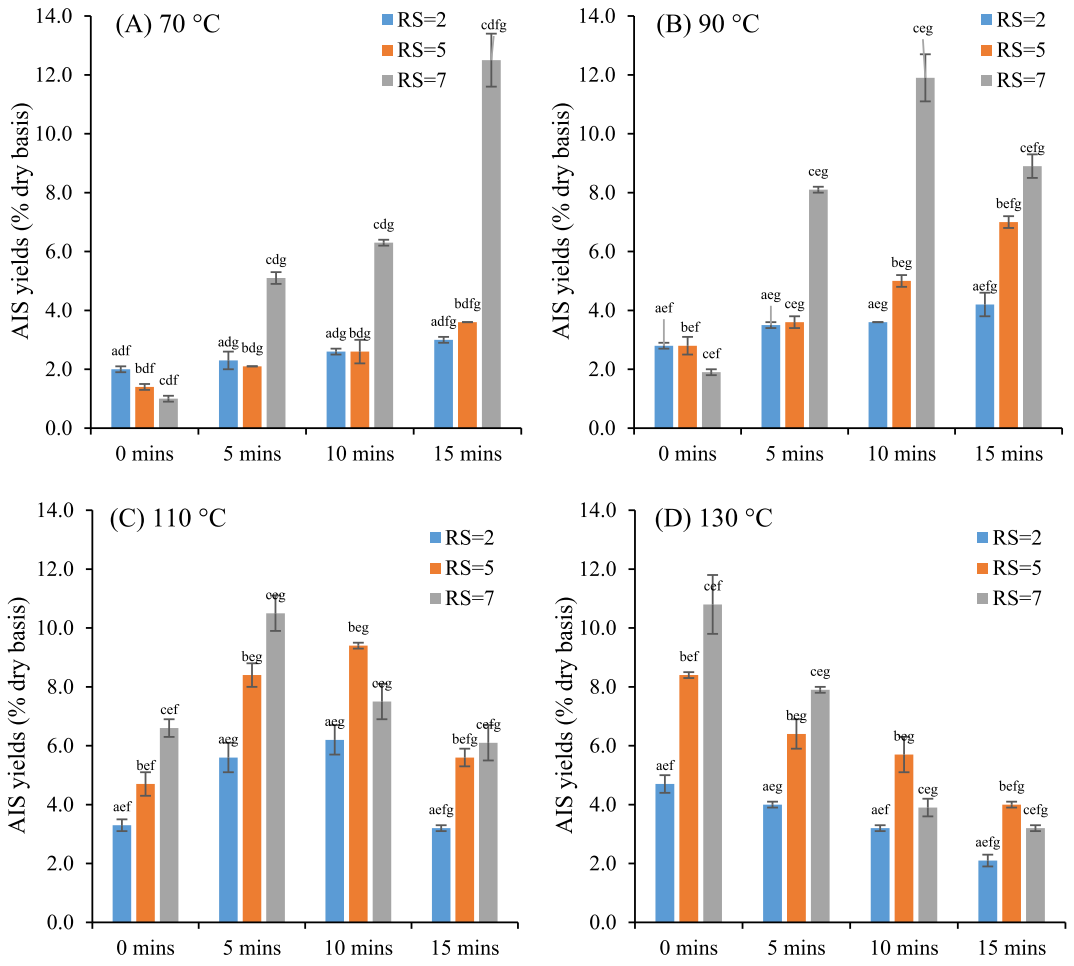

Fig. 2. AIS yields (% dry basis) comparing banana peels at different ripening stages (RS). AIS yields obtained at 0–15 min and (A) 70 °C, (B) 90 °C, (C) 110 °C and (D) 130 °C. Different letters in the same data figure indicate the significant difference ($p < 0.05$).

stage 1 to 5 and then decreased to 13.0% at stage 7, which is consistent with the AIS yield results from the present study at RS 1 to 5 but contrary from RS 5 to 7. In their work, AIS was sequentially extracted using water, chelator and acid in the named order with a total extraction time of 10 h, and the yields were not optimised. It is possible that RS 7 banana peel pectin started degrading at the prolonged condition and therefore yields decreased.

Apart from the abovementioned influence of ripening stages, extraction temperature and time also had significant influence of the AIS yields. Specifically when increasing the temperature to 130 °C, the extraction time reduced to 0 mins (0 mins is the holding time after the microwave system reached the desired temperature, refer to Section 2.2 for details) to obtain the highest AIS yields from all banana peel samples, exhibiting a potential for the design of a continuous extraction process. While increasing the extraction time to 15 or 10 mins, the highest AIS yields were achieved at 70 or 90 °C respectively, which are both below the solvent boiling point meaning an atmospheric pressure system design is sufficient for pectin extraction, presenting potentials to ease the safety concerns over pressurised system from a chemical engineering perspective. It is also worth noting that AIS yields decreased dramatically at higher temperatures and/or longer times, and this effect was more pronounced at higher the ripening stages. This might be due to the depolymerisation of banana peel pectin at higher ripening stages (as indicated by the low HGA and RG-I region contents in Table 1), which leads to the reduced molecular weight of the pectins preventing them from precipitating as AIS (Baghdadi, Nayebzadeh, Aminifar, & Mortazavian, 2023).

3.3. Extract chemical compositions

The results of the chemical and physical characterisation of the extracts are shown in Table 2. Pectin structure is primarily characterised by HGA and RG-I regions and the individual monosaccharides that made up the two regions. HGA is characterised by a GalA. backbone with methyl- and acetyl- side groups. RG-I is characterised by an alternating GalA. and Rha backbone with Ara. And Gal. side chains. Glu. and Xyl., represent starch and/or hemicellulose impurities in the AIS extract. The HGA and RG-I contents were calculated using Eq. (4) and (5). Fig. 3 shows the HGA and RG-I extractability, which represent the percentage amount of respective regions extracted from the total available amount in the raw banana peel material (% dry basis) as calculated based on Eq. (2) and (3). The characterisation results presented in Table 2 and Fig. 3 are discussed in the following sections to compare a). the effect of temperature (temperature varied from 70 °C to 130 °C and extraction time fixed at 5 mins); b). the effect of time (time varied from 0 to 15 mins and temperature fixed at 110 °C) and c). the effect of banana ripening (ripening stage 2,5, and 7 were studied).

3.3.1. HGA region pectin

HGA is a key parameter defining pectins and their applications. According to the specifications on purity characteristics of the Joint FAO/WHO Expert Committee on Food Additives and the European Commission, pectins should contain no <65% of HGA content (Muller-Maatsch et al., 2016). From the present study, the AIS extracts obtained from the highest yielding conditions had HGA contents higher than 65% (showing as ≥650 mg/g AIS) at all ripening stages indicating they can be

Table 2
Chemical compositions of AIS extracted at 70–130 °C, 0–15 min from banana peels at ripening stages 2, 5 and 7.

	GalA.	Rha.	Ara.	Gal.	Glu.	Xyl.	Total tested sugar	HGA region	RG-I region	HGA/RG-I ratio
	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	mg/g AIS	Weight ratio
Banana peel AIS at RS 2										
70 °C, 5mins	297.9 ± 1.6^{Bacef}	2.9 ± 0.2^{Cace}	24.9 ± 0.9^{Dacdg}	37.2 ± 1.1^{Eacf}	48.7 ± 2.1^{Fbcef}	12.1 ± 0.5^{Gadg}	423.7 ± 6.4^{Habde}	295.0 ± 1.8^{Iacef}	67.9 ± 2.4^{Jacg}	4.3 ± 0.2^{Kacef}
90 °C, 5mins	397.6 ± 4.2^{Badef}	2.6 ± 0.1^{Cace}	30.5 ± 1.2^{Daeg}	41.6 ± 1.6^{Eacf}	53.6 ± 2.7^{Fbdef}	12.7 ± 0.2^{Gaeg}	538.6 ± 10.0^{Hacde}	395.0 ± 4.3^{Iadef}	77.3 ± 3.0^{Jadg}	5.1 ± 0.4^{Kadef}
110 °C, 0mins	425.3 ± 3.7^{Badg}	3.4 ± 0.2^{Cade}	28.9 ± 0.4^{Dadef}	41.0 ± 1.9^{Eadf}	59.2 ± 2.9^{Fbde}	13.1 ± 0.3^{Gaefg}	570.9 ± 9.4^{Hace}	421.9 ± 3.9^{Iadg}	76.7 ± 2.7^{Jaef}	5.5 ± 0.4^{Kacdg}
110 °C, 5mins	587.5 ± 5.1^{Badef}	4.9 ± 0.4^{Cade}	35.7 ± 1.3^{Dadeg}	48.3 ± 1.1^{Eadf}	64.2 ± 3.0^{Fbdef}	13.4 ± 0.7^{Gaefg}	754.0 ± 11.6^{Hacde}	582.6 ± 5.5^{Iadef}	93.8 ± 3.2^{Jaeg}	6.2 ± 0.6^{Kacdef}
110 °C, 10mins	791.7 ± 8.9^{Badfg}	6.5 ± 0.7^{Cadf}	26.8 ± 1.3^{Dadeg}	51.3 ± 1.4^{Eadg}	78.7 ± 3.2^{Fbdg}	14.3 ± 0.4^{Gaefg}	969.3 ± 15.9^{Hace}	785.2 ± 9.6^{Iadfg}	91.1 ± 4.1^{Jaeh}	8.6 ± 0.9^{Kacdg}
110 °C, 15mins	522.6 ± 4.7^{Bade}	7.1 ± 0.6^{Cadf}	20.6 ± 0.4^{Dadef}	52.1 ± 2.1^{Eadg}	69.9 ± 2.6^{Fbdfg}	14.9 ± 0.6^{Gaefg}	687.2 ± 11.0^{Hacd}	515.5 ± 5.3^{Iade}	86.9 ± 3.7^{Jaeg}	5.9 ± 0.3^{Kacde}
130 °C, 5mins	449.5 ± 4.9^{Bacef}	5.8 ± 0.3^{Cade}	27.9 ± 0.7^{Dacg}	53.2 ± 1.6^{Eaef}	74.9 ± 2.2^{Fbdef}	16.2 ± 1.1^{Gafg}	627.5 ± 10.8^{Habde}	443.7 ± 5.2^{Iacef}	92.7 ± 2.9^{Jaeg}	4.8 ± 0.3^{Kabef}
Banana peel AIS at RS 5										
70 °C, 5mins	498.7 ± 2.2^{Bbcef}	3.6 ± 0.1^{Cbce}	29.9 ± 0.2^{Dbcdg}	39.1 ± 2.1^{Ebcf}	27.8 ± 1.3^{Facef}	13.1 ± 0.2^{Gbdg}	612.2 ± 6.1^{Habde}	495.1 ± 2.3^{Ibcef}	76.2 ± 2.5^{Jbcg}	6.5 ± 0.5^{Kacef}
90 °C, 5mins	626.1 ± 3.5^{Bbdef}	4.2 ± 0.2^{Cbce}	36.2 ± 0.9^{Dbeg}	42.8 ± 2.3^{Ebcf}	41.2 ± 2.6^{Fadef}	15.2 ± 0.4^{Gbeg}	765.7 ± 9.9^{Hacde}	621.9 ± 3.7^{Ibdef}	87.4 ± 3.6^{Jbdg}	7.1 ± 0.6^{Kadef}
110 °C, 0mins	673.0 ± 4.1^{Bbdg}	5.4 ± 0.2^{Cbde}	20.1 ± 0.2^{Dbdef}	49.2 ± 1.9^{Ebdf}	34.9 ± 2.1^{Fade}	15.0 ± 0.4^{Gbefg}	797.6 ± 8.9^{Hace}	667.6 ± 4.3^{Ibdg}	80.1 ± 2.5^{Jbef}	8.3 ± 0.9^{Kacdg}
110 °C, 5mins	813.4 ± 6.9^{Bbdef}	6.1 ± 0.4^{Cbde}	41.9 ± 1.1^{Dbdeg}	51.3 ± 2.8^{Ebdf}	46.8 ± 1.9^{Fadef}	16.0 ± 1.0^{Gbefg}	975.5 ± 14.1^{Hacde}	807.3 ± 7.3^{Ibdef}	105.4 ± 4.7^{Jbeg}	7.7 ± 0.8^{Kacdef}
110 °C, 10mins	645.9 ± 5.4^{Bbdfg}	7.6 ± 0.5^{Cbdf}	37.9 ± 0.6^{Dbdeg}	55.3 ± 3.1^{Ebdg}	52.6 ± 3.0^{Fadg}	17.1 ± 0.8^{Gbefg}	816.4 ± 13.4^{Hace}	638.3 ± 5.9^{Ibdfg}	108.4 ± 4.7^{Jbeh}	5.9 ± 0.4^{Kacdf}
110 °C, 15mins	529.6 ± 3.8^{Bbde}	7.4 ± 0.4^{Cbdf}	31.6 ± 0.8^{Dbdef}	56.2 ± 3.4^{Ebdg}	49.5 ± 2.4^{Fadfg}	16.2 ± 0.7^{Gbefg}	690.5 ± 11.5^{Hacd}	522.2 ± 4.2^{Ibde}	102.6 ± 5.0^{Jbeg}	5.1 ± 0.2^{Kacde}
130 °C, 5mins	432.8 ± 3.7^{Bbcef}	7.9 ± 0.4^{Cbde}	28.2 ± 0.4^{Dbcg}	59.9 ± 2.9^{Ebef}	50.1 ± 3.7^{Fadef}	17.4 ± 1.2^{Gbfg}	596.3 ± 12.3^{Habde}	424.9 ± 4.1^{Ibcef}	103.9 ± 4.1^{Jbeg}	4.1 ± 0.2^{Kabef}
Banana peel AIS at RS 7										
70 °C, 5mins	449.4 ± 3.2^{Bacef}	4.3 ± 0.3^{Cace}	31.6 ± 1.1^{Dbcdg}	40.5 ± 3.0^{Eabcf}	54.3 ± 4.1^{Facef}	15.4 ± 0.1^{Gcdg}	595.5 ± 11.8^{Habde}	445.1 ± 3.5^{Iacef}	80.7 ± 4.7^{Jbcg}	5.5 ± 0.2^{Kacef}
90 °C, 5mins	738.5 ± 4.7^{Badef}	4.7 ± 0.4^{Cace}	37.4 ± 0.8^{Dbeg}	41.2 ± 2.8^{Eabcf}	69.8 ± 4.4^{Fadef}	16.9 ± 0.3^{Gceg}	908.5 ± 13.4^{Hacde}	733.8 ± 5.1^{Iadef}	88 ± 4.4^{Jbdg}	8.3 ± 0.4^{Kadef}
110 °C, 0mins	842.1 ± 6.1^{Badg}	4.9 ± 0.6^{Cade}	33.9 ± 0.9^{Dbdef}	40.8 ± 2.2^{Eabdf}	57.6 ± 2.9^{Fade}	17.1 ± 0.2^{Gcefg}	996.4 ± 12.9^{Hace}	837.2 ± 6.7^{Iadg}	84.5 ± 4.3^{Jbef}	9.9 ± 0.6^{Kacdg}
110 °C, 5mins	539.9 ± 2.9^{Badef}	5.6 ± 0.9^{Cade}	47.5 ± 1.2^{Dbdeg}	52.4 ± 2.9^{Eabdf}	76.1 ± 3.9^{Fadef}	17.6 ± 0.9^{Gcefg}	739.1 ± 12.7^{Hacde}	534.3 ± 3.8^{Iadef}	111.1 ± 5.9^{Jbeg}	4.8 ± 0.2^{Kacdef}
110 °C, 10mins	386.2 ± 2.0^{Badfg}	5.4 ± 0.4^{Cadf}	40.2 ± 1.4^{Dbdeg}	51.1 ± 3.5^{Eabdg}	73.2 ± 5.6^{Fadg}	17.9 ± 1.3^{Gcefg}	574.0 ± 14.2^{Hace}	380.8 ± 2.4^{Iadfg}	102.1 ± 5.7^{Jbeh}	3.7 ± 0.3^{Kacdf}
110 °C, 15mins	216.8 ± 1.5^{Bade}	4.1 ± 0.2^{Cadf}	29.8 ± 0.6^{Dbdef}	50.2 ± 3.8^{Eabdg}	69.3 ± 2.2^{Fadfg}	17.2 ± 0.5^{Gcefg}	387.4 ± 8.8^{Hacd}	212.7 ± 1.7^{Iade}	88.2 ± 4.8^{Jbeg}	2.4 ± 0.1^{Kacde}
130 °C, 5mins	207.4 ± 1.6^{Bacef}	3.2 ± 0.3^{Cade}	27.3 ± 0.4^{Dbcg}	53.6 ± 3.1^{Eabef}	64.2 ± 4.0^{Fadef}	16.4 ± 0.6^{Gcfg}	372.1 ± 10.0^{Habde}	204.2 ± 1.9^{Iacef}	87.3 ± 4.1^{Jbeg}	2.3 ± 0.1^{Kabef}

Different letters in the same data column indicate the significant difference ($p < 0.05$).

classified as a good source of pectin. The highest HGA content of 837.2 mg/g extract were achieved at 110 °C, 0 mins from RS 7, followed by 807.3 mg/g AIS at 110 °C, 5 mins from RS 5, and 785.2 mg/g AIS at 110 °C, 10 mins from RS 2 (shown in Table 2). The observed results can be explained by the conversion of pectin during the fruit ripening. At the early ripening stage, pectin is predominantly present in the form of protopectin, which is a water-insoluble and high molecular weight parent pectin, and thus less extractable by the implemented extraction method using water in this work (Chen et al., 2019). As ripening progresses, this tightly bound protopectin is degraded into soluble pectin (in the form of polysaccharides) and finally pectic acids (mainly polyuronic acids), which are found loosely bound to the cell walls and more available to be extracted (Prasanna et al., 2007). Those results comparing ripening stages were also in accordance with the literature, for example, Castillo-Israel et al. (2015) reported an increase of pectin HGA region content from 397 mg/g AIS to 573 mg/g AIS extracted respectively from unripe to ripe banana peels using acids as the solvents. AIS extracts from other extraction conditions had HGA content <650 mg/g AIS, which indicated impurities possibility due to the presence of proteins, starch and hemicellulose etc. (Castillo-Israel et al., 2015).

The HGA region extractability (shown in Fig. 3A-3B) varied from 1.5% to 27.3%, and were generally higher in extracts from higher ripening stages across the tested extraction conditions despite the fact that the total HGA content in the banana peel raw material reduced during ripening (results from Section 3.1). In addition, the highest extractability results were achieved under milder extraction conditions (i.e. lower temperature or shorter time). For example, extracts from RS 7 obtained the highest extractability of 27.3% at 90 °C, 5 mins, while 110 °C and 10 mins were required to achieve the maximum extractability of 10.4% at RS2. After those condition, the HGA extractability decreased quickly. These results were similar to the AIS yield results (Section 3.2) and they can be explained by the increased solubility of the depolymerised HGA-rich pectin by the abovementioned protopectin conversion mechanisms during the fruit ripening.

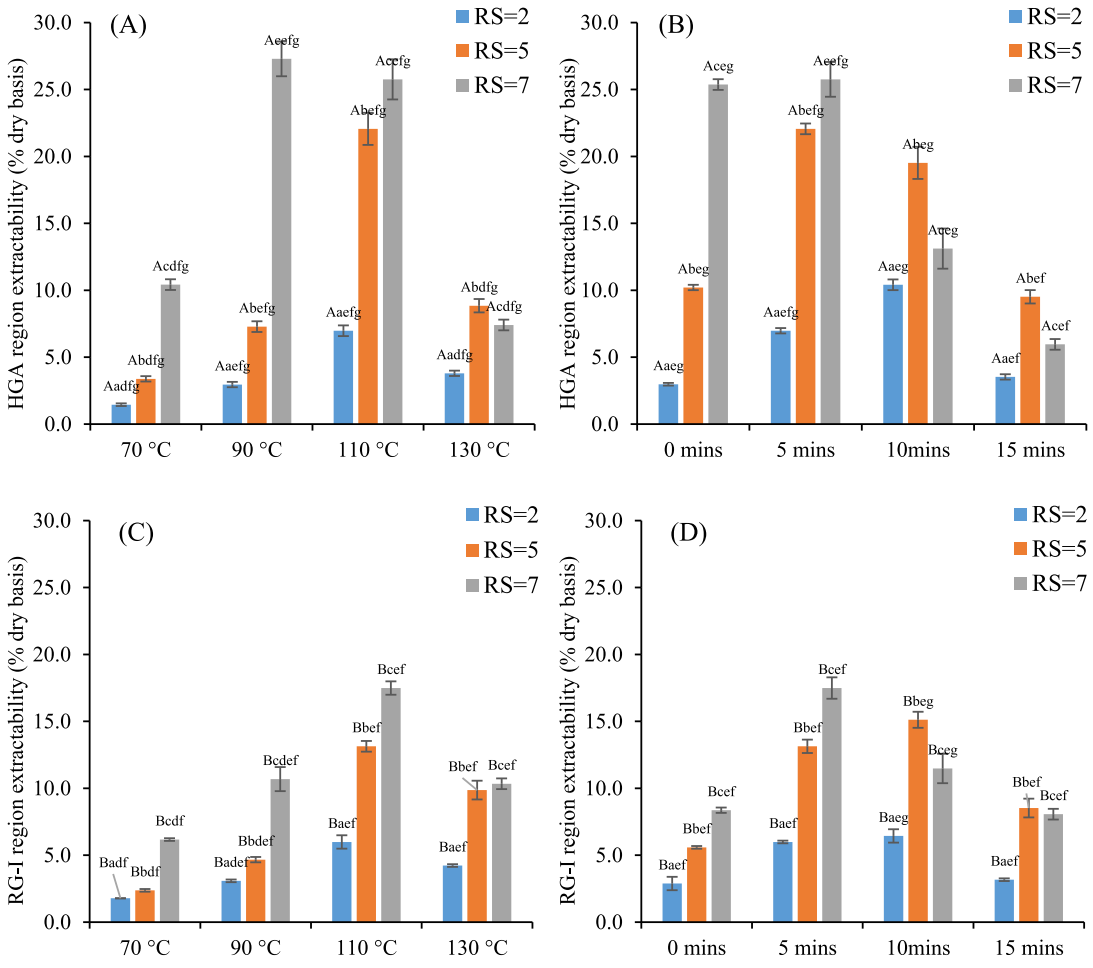

Fig. 3. HGA and RG-I region extractability (% dry basis) for AIS from banana peels at ripening stages 2, 5 and 7. (A) HGA region extractability at 70–130 °C, 5 mins; (B) HGA region extractability at 0–15 min, 110 °C; (C) RG-I region extractability at 70–130 °C, 5 mins; (D) RG-I region extractability at 0–15 min, 110 °C. Different letters in the same data figure indicate the significant difference ($p < 0.05$).

3.3.2. *RG-I region pectin*

The contents of RG-I region and its constituent monosaccharides (mainly Rha, Ara. and Gal.) varied with the extraction conditions and ripening stages. The highest RG-I region contents were achieved at 111.1 mg/g AIS at RS 7, and decreased to 108.4 mg/g AIS at RS 5 and 91.1 mg/g AIS at RS 2. Those results indicate that although the RG-I region contents in the raw material generally decreased during ripening (results discussed in Section 3.1), the RG-I region became loosened from the cell wall during ripening (Emaga, Robert, et al., 2008). This may be attributed to the enzymatic action of cell wall hydrolases (such as glucanase) to break the in vivo binding between cellulosic glucan and RG-I side chains (Broxterman & Schols, 2018; Zykwinska, Rondeau-Mouro, Garnier, Thibault, & Ralet, 2006). Additionally, although the RG-I became more available to extract, the responses of the three monosaccharides were slightly different; the extraction of Rha. and Gal. were favoured at higher temperatures and times, while Ara. extraction reached its highest at lower temperature and time.

The highest RG-I extractability results (shown in Fig. 3C-3D) were achieved at the same conditions as for RG-I contents. The highest RG-I extractability decreased from 17.5% at RS 7 to 15.1% at RS 5 and 6.4% at RS 2. A similar trend of RG-I region extractability, which decreased from 8.2% to 5.2% at RS 7 and 5 respectively can be found in the work of Emaga, Robert, et al. (2008). A dramatic increase to 15.6% at RS 1 was reported; however, the results comparing the different banana materials were reported from the same extraction condition (80 °C and 1 h in dilute HCl solution), which may not be its optimum condition (Emaga, Robert, et al., 2008). It is worth noting that the higher contents of RG-I were obtained at longer time and/or higher temperature than HGA region. This suggests that the RG-I region is harder to extract, and this is attributed to the multiple side chains in the RG-I region resulting in a higher molecular weight and a more complex molecular configuration (Mao et al., 2020).

3.4. *DM and DA*

The DM and DA are key parameters determining HGA pectin functionality including gelling ability, surface activity, emulsion stability, and viscosity, and thus having huge impact on the extract application (Müller-Maatsch, Caligiani, Tedeschi, Elst, & Sforza, 2014). As shown in Fig. 4A-4B, the DM results in the presented study varied across banana peel ripening stages and the extraction conditions from 21.0% to 82.6%, which were within the ranges reported in the literature (Emaga, Robert, et al., 2008). The DM values were both influenced by the ripening stages of the raw banana peel material and the extraction conditions. At RS 7, the highest DM of 82.6% was obtained at 110 °C and 10 mins; after which it decreased at higher temperature and/or longer extraction time. The DM at RS 2 and 5 continued to increase at all tested temperature and time range and reached its highest values of 67.1% and 73.1% respectively both from 110 °C and 10 mins. Similar results comparing the banana peel ripening stages were reported by Emaga, Robert, et al. (2008), where the DM increased from 35.0% at RS 1 to 44.0% at RS 5

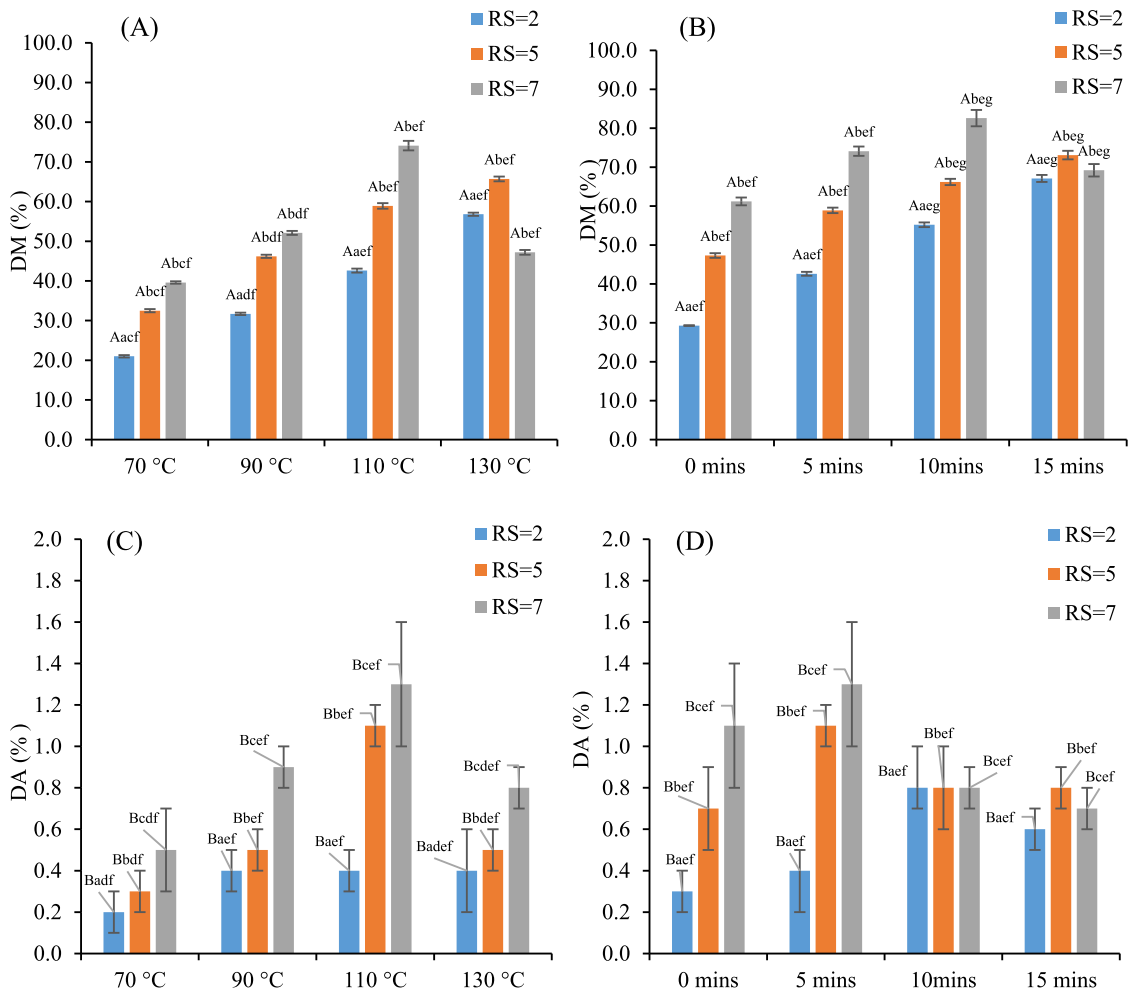

Fig. 4. Degree of methylation (DM, %) and acetylation (DA, %) for AIS from banana peels at ripening stages 2, 5 and 7. (A) DM at 70–130 °C, 5 mins; (B) DM at 0–15 min, 110 °C; (C) DA at 70–130 °C, 5 mins; (D) DA at 0–15 min, 110 °C. Different letters in the same data figure indicate the significant difference ($p < 0.05$).

and 67.1% at RS 7. Kamble, Gawande, and Patil (2017) compared the effect of extraction conditions on unripe banana peels and found the increase of DM from 75.95% to 80.18% with the increase of extraction time from 1 to 4 h. It is generally believed that the DM of pectic polysaccharides in the cell wall reduces with ripening catalysed by the presence of pectin methyl esterase (PME), which is the first enzyme to act on pectin during the fruit ripening (Kohli, Kalia, & Gupta, 2015). This de-methylesterified pectin exhibits increased enzymatic amenability and undergoes quicker degradation into smaller molecular weight pectic acids under the continued action of other enzymes such as polygalacturonase (PG). Pectin that is less influenced by the action of PME tends to retain its DM and structural integrity (Giovane et al., 2004). This part of higher DM pectin exhibits better water solubility due to the presence of methyl groups and low alcohol solubility due to its relatively higher molecular weight (Gawkowska, Cybulska, & Zdunek, 2018), and thus is preferentially extracted into the water solvent and then precipitated into the AIS fraction as in the presented method. This may explain the increasing DM in the AIS extracts in the present work and other abovementioned research, despite the general consensus in the literature that the DM of protopectin decreases on ripening. However, more work to characterise the DM of the alcohol-soluble fraction (ASF, the supernatant after alcohol addition) is required to further elucidate this, which is not within the scope of the present study. The DA values varied from 0.2% to 1.3% (Fig. 4C-4D), which was also consistent with the results from the literature (Emaga, Robert, et al., 2008); however, the changes were less conclusive taking into account the error bars. The results for DA were much lower than DM suggesting the negligible impact of DA on the banana peel extracts, and therefore it was not further discussed here.

3.5. Intrinsic viscosities

The intrinsic viscosity can provide information relating to the size and hydrodynamic volume of polysaccharides (such as pectin) and fundamental properties of the polysaccharides and its interaction with solvent, which influence the functionality and application of the polymer (Ako, Elmarhoum, & Munialo, 2022). The intrinsic viscosity results of banana peel AIS (Fig. 5) varied between 94 and 309 mL/g and was within the range reported by the literature (Emaga, Robert, et al., 2008), although they were lower than other biomasses such as orange peel (522 mL/g (Mao et al., 2020)) and mango peel (320–1346 mL/g (Koubala et al., 2008)). Results show that both the ripening stage of the raw banana peel material and the extraction conditions had significant influence on the intrinsic viscosity; more specifically, the highest intrinsic viscosity values of AIS from higher ripening stage banana peels usually occurred at milder extraction conditions (lower temperatures or shorter times) than those from less ripe peels. At RS 7, the highest intrinsic viscosity of 291 mL/g was obtained at 110 °C and 0 mins; after which it decreased at higher temperature and/or longer extraction time, while at RS 2, it required 110 °C and a longer 10 mins to reach the comparable value of 261 mL/g. Emaga, Robert, et al. (2008) reported the increase of intrinsic viscosity of water-extracted pectin with the

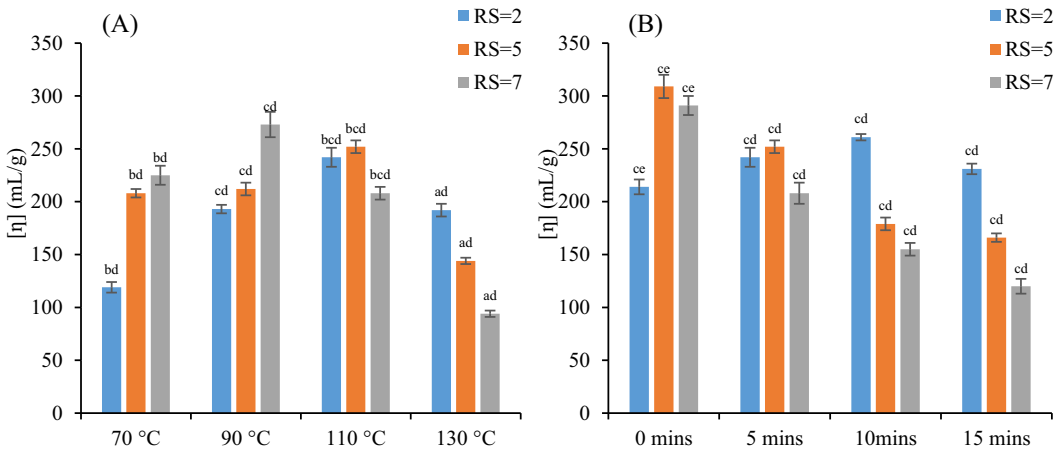

Fig. 5. Intrinsic viscosity ([η], mL/g) for AIS from banana peels at ripening stages 2, 5 and 7. (A) 70–130 °C, 5 mins; (B) 0–15 min, 110 °C. Different letters in the same data figure indicate the significant difference ($p < 0.05$).

increase of ripening stages of banana peel, but the results varied when using other solvents. Most literature reported the decrease of intrinsic viscosity with the stronger extraction condition (Kaya et al., 2014), but others reported the opposite (Momeni et al., 2018). The intrinsic viscosity can often be correlated with the molecular weight by the Mark-Houwink-Sakurada equation (Jong, Abdullah, & Muhammad, 2023):

$$[\eta] = kM_v^{\alpha} \quad (9)$$

Where, [η] is the intrinsic viscosity (mL/g); M_v is the viscosity-average molecular weight (g/mol); and k and α are constants for a given solute-solvent system and temperature. However, for complex polysaccharide polymers (such as pectin), *the* intrinsic viscosity may be not only affected by the molecular weight, but also various factors including polymer rigidity and configuration as well as the strength of hydrogen bonding (Momeni et al., 2018). For example, pectin with higher RG-I content usually exhibits higher molecular weight due to the presence of numerous side chains in RG-I region compared with the HGA region (Mao et al., 2020). However, HGA-rich pectins are often more viscous due to their ability to form emulsions in the solvents (Ngouémazong, Christiaens, Shpigelman, Van Loey, & Hendrickx, 2015), which also varies depending on the DM (Jong et al., 2023). Future work is required to investigate the polymer structure and configuration and to gain fundamental understanding of this complex polymer's hydrodynamics.

4. Implications and future work on the development of banana peel pectin extraction and application for banana peel valorisation

Overall, the results demonstrated the efficiency of the presented method in banana peel pectin extraction in achieving high AIS yields with both HGA and RG-I region preserved and a wide range of DM. This work provided a sustainable solution for banana peel valorisation and the potential for banana peel pectins in various applications. Specifically, ripened banana peel (RS 7) was found promising for pectin extraction.

The most common reported applications of banana peel pectin from the literature are as traditional gelling agents or emulsifiers (Rasidek et al., 2021), which was closely related to the chemical compositions (i.e. HGA and RG-I region) and DM. The functionality of pectin to gel is dependent on its HGA and RG-I region content, in that a low amount of RG-I (HGA/RG-I ratio > 2) could improve long term stability of emulsions as opposed to the pectin that contained a high amount of RG-I region (Rekpodo et al., 2018). All the banana peel AIS extracts obtained at the highest yielding condition in this work were HGA-rich pectins with a relatively low amount of RG-I, indicating the potential suitability of our extracts at all three tested ripening stages as gelling agents. Specifically, pectins from ripened banana peels at RS 7 exhibit the highest potential for gelling application compared to pectins from lower ripening stages as they contain the highest HGA region content of 842.1 mg/g AIS and highest HGA/RG-I ratio of 9.9. In addition, these potentially high-gelling pectins from RS 7 are able to be recovered at a shorter extraction time of 0 mins (and at 110 °C), suggesting the potential for the design of a continuous extraction process. However, future work to test the gelling properties of the extracts is required for further product development. DM is another factor influencing pectin gelling ability. Depending on the DM, pectins are classified as high-methyl pectin (HMP, with DM above 50%) and low-methyl pectin (LMP, with DM below 50%). HMP and LMP have different conditions and mechanisms of gelling, i.e., HMP forms gel in the presence of high amount of sugar (55–85%); while LMP requires divalent ions to form gel (Maneerat, Tangsuphoom, & Nitithamyong, 2017). From the presented work, HMP can be obtained from banana peels at higher ripening stages, at higher extraction temperature and longer times; while vice versa for LMP. The results show the potential to manipulate the pectin functionality and therefore application by selecting the raw banana peel materials at different ripening stages. However, more work is required to prove the extract functionality, including but not limited to particle molecular weight, rheological characterisation, emulsifying characterisation and thermal analysis (Dranca & Oroian, 2018).

Banana peel AIS obtained in this work may also be suitable for novel and value-added applications such as prebiotics, which is closely connected with the pectin RG-I region. Raw banana peel is not traditionally considered a good source of RG-I pectin because it contains less RG-I region content (66.7–87.7 mg/g biomass as reported in the presented study) than other biomasses sources, such as sugar beet (240 mg RG-I region pectin/g biomass (Mao et al., 2019)) or potato peels (366 mg RG-I region pectin /g biomass (Khodaei & Karboune, 2013)). However, the banana peel pectin extracts obtained in this present study had comparable RG-I compositions to pectin extracted from other biomass sources as reported in the literature, and ripened banana peel was found more favourable to produce pectins with higher RG-I region contents under milder extraction conditions (lower temperature and shorter time). Gómez et al. (2016) reported the promising prebiotic ability of pectin extracts from sugar beet pulp (HGA region content = 404.9 and RG-I region content = 139.5 mg/g AIS) and lemon peel (HGA region content = 521.1 mg/g and RG-I region content =102.1 mg/g AIS). In comparison, our banana peel pectin extract obtained under 110 °C, 5 mins from RS 7 banana peel had a very similar chemical composition consisting of 534.3 mg/g of HGA and 111.1 mg/g of RG-I. This implies the potential application of banana peel pectins from this study as prebiotics, although future work, for example in vitro fermentability assessment and immunological study, is required to elucidate this.

Additionally, the information of the antioxidant and antimicrobial properties would also be a beneficial addition to expand extracts applications in novel and value-added realms.

There are several advantages associated with the implemented extraction method. It only used water as the solvent due to its green and sustainable nature. Unlike traditional banana peel pectin extraction that often includes acids (Rivadeneira et al., 2020), water not only helps to preserve the pectin structure to allow potentially expanded applications (as discussed above), it is food-grade meaning the products do not require purification nor the solvents need further treatment before disposal. The extractions can reach highest yields in only 5–10 min and potentially be reduced to 0 mins when applying a higher temperature (i.e. 130 °C). This will allow the design of a continuous system; however, further work needs to be completed to understand pectin extraction and degradation kinetics to achieve this. The yield results also suggest that milder extraction conditions (i.e. lower temperature and/or shorter time) and potentially less energy are needed for ripened banana materials. The yield results decreased dramatically after the optimum conditions, which may be due to the pectin degradation during the thermal processing. The fundamental mechanism of the decrease in yield after optimum condition was not studied in this manuscript and further work is required to elucidate this. However, microwave shows its advantage in this case as it allows quicker heating and better temperature control compared with conventional heating (Mao et al., 2023).

5. Conclusion

This paper reports a study to investigate a sustainable and efficient approach of microwave-assisted pectin extraction for banana peel waste valorisation. The novelty of this work is the investigation of the influence of banana ripening stage on pectin extraction and the investigation of a feasible route to recover both HGA and RG-I region from banana peel. The banana ripening showed a positive effect on pectin extraction for both HGA and RG-I regions. The highest AIS yield of 12.5% was achieved at 70 °C, 15 mins from RS 7 peel. Further chemical characterisations showed increased HGA extractability (from 10.4% at RS2 to 27.3% at RS7), RG-I extractability (from 6.4% for RS 2 to 17.5% at RS 7) as well as increased DM (from 67.1% at RS2 to 82.6% at RS7). These results suggest the pectin depolymerisation and cell wall cleavage during banana ripening, which could be attributed to a range of working enzymes, make pectin more available to extract at higher ripening stages. The results also suggest that milder extraction conditions (i.e. lower temperature and shorter time) and potentially less energy are needed for ripened banana materials. The chemical compositions of the banana peel AIS obtained in this work suggest their potential applications as not only traditional gelling agent but also novel value-added applications such as prebiotics; however, more work is required to elucidate this.

CRediT authorship contribution statement

Yujie Mao: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Writing – original draft. **Shinta R. Dewi:** Formal analysis. **Stephen E. Harding:** Methodology, Supervision. **Eleanor Binner:** Conceptualization, Funding acquisition, Project administration, Resources, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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