

Article

Multifunctional Citrus Peel Pectins from Seven Species: A Comparative Study of Physicochemical, Techno-Functional, and Bioactive Properties

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Abstract

Citrus peel, a major by-product of citrus processing, is a rich source of pectin and other bioactive compounds. In this study, pectin extracted from seven citrus species was comprehensively characterized for its structural, spectral, thermal, physicochemical, techno-functional, antioxidant, and anti-amylase properties. The extracted pectin (~50% methyl esterification) contained residual cellulose, hemicellulose, lignin, and proteins, with Fourier-transform infrared (FTIR) and ultraviolet-visible (UV-Vis) confirming typical galacturonic acid-based structures. Fluorescence analysis revealed emission shifts (420–500 nm) compared to standard pectin. Thermal analysis indicated multi-stage degradation, with major transitions linked to moisture loss, polysaccharide depolymerization, and lignin oxidation. X-ray diffraction (XRD) confirmed a predominantly amorphous structure with minor cellulose domains. The pectin solution showed negative zeta potential, shear-thinning behavior, and high conductivity. Functionally, it exhibited strong swelling, hygroscopicity, and water/oil holding capacity, but low foaming ability. Biologically, it demonstrated moderate antioxidant activity and α -amylase inhibition. These findings highlight citrus pectin as a promising sustainable ingredient for food and pharmaceutical applications, with future work needed to enhance its solubility and bioactivity.

Keywords: citrus peel pectin; bioactive compounds; techno-functional properties; antioxidant activity; amylase inhibition



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1. Introduction

Citrus represents the most extensively cultivated fruit group in the global agro-industrial sector, serving as a prolific source of nutritionally and therapeutically significant compounds. These include flavonoids, terpenes, carotenoids, and essential oils, all of which find broad utility in the food and pharmaceutical industries [1,2]. Despite this, a substantial volume of citrus biomass is currently relegated to low-value applications, such as animal feed or composting. Recent research underscores a critical opportunity for “waste-to-wealth” conversion, particularly through the valorization of citrus peel, the largest by-product stream of processing. This biomass can be refined into high-value pectin, proteins, and bioactive phytochemicals for use in the nutraceutical and pharmaceutical sectors [3–5].

Pectin is a complex structural polysaccharide within the primary cell wall, predominantly composed of homogalacturonan (HG) and rhamnogalacturonan (RG) regions. Its

functionality is largely regulated by varying levels of esterification, which influence cell adhesion, wall porosity, and tissue integrity [6]. Beyond its structural role, pectin modulates developmental stages, including fruit ripening and organ abscission, through enzymatic remodeling by pectin methylesterases. Furthermore, the partial hydrolysis of pectin generates oligogalacturonides; these molecules function as Damage-Associated Molecular Patterns (DAMPs), which trigger innate immunity and activate plant defense pathways against pathogens [7].

In human nutrition, pectin functions as a versatile soluble dietary fiber. By forming high-viscosity solutions, it slows gastric emptying and glucose absorption, thereby attenuating postprandial glycemia. Upon reaching the colon, pectin is fermented by gut microbiota into short-chain fatty acids (SCFAs), acetate, propionate, and butyrate, which support gut barrier integrity and immune regulation [8,9]. Moreover, clinical evidence consistently supports its cardiovascular benefits, particularly its cholesterol-lowering effects mediated through bile acid binding [10].

Modified Citrus Pectin (MCP) exhibits unique bioactivities beyond standard fiber effects, notably its ability to bind Galectin-3, a lectin associated with fibrosis, inflammation, and tumor progression. Consequently, MCP is being investigated for its potential to inhibit metastasis and modulate chronic inflammatory pathways. Additionally, pectin's chelating properties allow it to bind heavy metals like lead and mercury, facilitating their detoxification [11]. Owing to its biocompatibility and mucoadhesive properties, pectin is also an ideal candidate for colon-targeted drug delivery. Its resistance to upper gastrointestinal enzymes enables the site-specific release of anti-inflammatory agents, probiotics, and chemotherapeutics [12].

Recent innovations in nanotechnology have introduced pectin-based nanocarriers, hydrogels, and composite biomaterials that provide superior encapsulation efficiency and controlled release, showing great promise for precision medicine and tissue engineering [13]. Despite the industrial ubiquity of citrus pectin, systematic comparative studies across different species remain scarce. This study systematically evaluates the techno-functional, thermal, and bioactive properties of pectins derived from seven distinct varieties: bitter orange (*C. aurantium*), lime (*C. limetta*), lemon (*C. limon*), pomelo (*C. maxima*), mandarin (*C. reticulata*), grapefruit (*C. paradisi*), and sweet orange (*C. sinensis*). By identifying source-specific variations, this work aims to establish a foundational reference for the selective utilization of citrus varieties in tailored food stabilization and glycemic-control formulations. In contrast to previous studies that primarily focus on modified citrus pectin or single-source systems, this work provides a comprehensive comparative analysis across multiple citrus species, integrating structural, physicochemical, techno-functional, antioxidant, and anti-amylase properties within a unified framework. This holistic approach reveals previously underexplored relationships between pectin source and functionality, offering new insights for the rational design of citrus-derived ingredients and positioning these materials as versatile and tunable candidates for advanced food and biomedical applications.

2. Materials and Methods

2.1. Citrus Peel Preparation

Citrus fruits, including bitter orange (*Citrus aurantium*), lime (*C. limetta*), lemon (*C. limon*), pomelo (*C. maxima*), mandarin (*C. reticulata*), grapefruit (*C. paradisi*), and sweet orange (*C. sinensis*), were harvested in December from mature, healthy trees at the Fars Research Center for Agriculture and Natural Resources, Jahrom, Iran (28°30'48" N, 53°37'33" E; altitude 1070 m). Only sound, mold-free fruits were selected to minimize contamination. The fruits were washed with distilled water, treated with 0.1%

sodium bicarbonate solution for 10 min to remove surface residues, and rinsed again with distilled water. The peels, including both flavedo and albedo, were manually separated and treated with 10 mM EDTA for 10 min to chelate potential heavy metals, followed by thorough rinsing with deionized water. The cleaned peels were then shade-dried under ambient conditions with proper aeration, ground using a laboratory blender, sieved (100 mesh, $\sim 150\ \mu\text{m}$), and stored in sealed containers at $4\ ^\circ\text{C}$ until further analysis [14].

2.2. Preparation of Pectin from Whole Citrus Peel

Ten grams of citrus peel powder were mixed with 300 mL of 50 mM hydrochloric acid and stirred at room temperature for 30 min, followed by microwave-assisted extraction for 2 h. The mixture was then filtered and cooled to room temperature. Pectin was precipitated by adding 95% ethanol at a 1:2 (*v/v*) ratio and incubating at $4\ ^\circ\text{C}$ overnight. The precipitate was collected, washed three times with 95% ethanol, and dried at $30\ ^\circ\text{C}$ for 24 h [15]. The insoluble residue was recovered by filtration, air-dried overnight under a fume hood, frozen in liquid nitrogen, and lyophilized. This residue was used to determine the composition of major structural components, including pectin (m-hydroxydiphenyl assay), hemicellulose (phenol-sulfuric acid method), cellulose (Updegraff method), lignin (Klason method using 72% H_2SO_4), and protein (Bradford assay).

2.3. FTIR Spectroscopy of Citrus Pectin

Prior to FTIR analysis, citrus pectin powders were sieved through a 100-mesh screen to obtain a uniform particle size ($\sim 150\ \mu\text{m}$). Approximately 1 mg of each sample was placed directly onto the diamond crystal of the ATR accessory and gently compressed to ensure optimal contact. Spectra were recorded using a Bruker ALPHA II FTIR spectrometer (Bruker Optik GmbH, Ettlingen, Germany) equipped with an ATR unit over the range of $4000\text{--}400\ \text{cm}^{-1}$ at room temperature, with a resolution of $4\ \text{cm}^{-1}$. Background spectra were collected prior to each measurement [16]. Functional groups were identified based on characteristic absorption bands. The degree of methyl esterification (DM) was determined from the FTIR spectra by calculating the ratio of the integrated peak area at $1730\ \text{cm}^{-1}$ (esterified carboxyl groups) to the sum of the peak areas at $1730\ \text{cm}^{-1}$ and $1630\ \text{cm}^{-1}$ (total carboxyl groups), following established methodology [17]:

$$\text{DM (\%)} = 100 \times \frac{A_{1730}}{A_{1730} + A_{1630}}$$

where A_{1730} and A_{1630} represent the integrated areas of the absorbance bands for esterified and free carboxyl groups, respectively.

2.4. UV-Vis Absorption and Fluorescence Spectroscopy of Citrus Pectin

UV-Vis spectra (200–900 nm) were recorded using a Shimadzu UV-1280 spectrophotometer (Shimadzu Corporation, Kyoto, Japan). Fluorescence emission spectra were obtained with a Varian Cary Eclipse spectrofluorometer (Agilent Technologies, Santa Clara, CA, USA) at an excitation wavelength of 280 nm, with emission recorded from 290 to 700 nm. For analysis, a 1.0% (*w/v*) citrus pectin solution was prepared by dispersing 1.0 g of powder in 100 mL of distilled water. The pH was adjusted to 8.0 using ethanolamine to facilitate complete solubilization of pectin complexes [18].

2.5. Thermal Analysis of Citrus Pectin

Thermogravimetric analysis (TGA), derivative thermogravimetry (DTG), and differential scanning calorimetry (DSC) were conducted using a simultaneous thermal analyzer (SDT 650, TA Instruments, New Castle, DE, USA) to investigate the thermal behavior of citrus pectins. Approximately 10 mg of each dried sample was placed in an open alumina

crucible and heated from room temperature to 800 °C at a heating rate of 10 °C min^{−1} under an air atmosphere. TGA and DTG curves were used to evaluate mass-loss patterns and determine the corresponding degradation temperatures, while DSC analysis was employed to identify thermal transitions associated with moisture evaporation and pectin decomposition. These complementary techniques provided detailed information on the thermal stability and decomposition characteristics of the extracted pectins.

2.6. XRD Analysis of Citrus Pectin

The crystalline characteristics of citrus pectins were analyzed by XRD using a Bruker D8 Advance diffractometer (Bruker AXS, Karlsruhe, Germany) with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 30 mA. Diffraction patterns were recorded over a 2θ range of 10–80° at a scanning rate of 2° min^{−1}. The resulting patterns were used to evaluate crystallinity and distinguish between crystalline and amorphous regions. Phase identification was performed by comparison with standard reference patterns from the JCPDS database [19].

2.7. Physico-Chemical Properties of Citrus Pectin

Physicochemical analyses were performed to evaluate the colloidal stability and functional properties of citrus pectin dispersions. Electrical conductivity and osmolarity were measured to assess ionic strength, while zeta potential indicated electrostatic stability. Particle size distribution, viscosity, and surface tension were determined to characterize dispersion behavior, rheological properties, and interfacial performance. For analysis, a 1.0% (*w/v*) pectin solution was prepared by dispersing 1 g of peel powder in 100 mL of distilled water, as described above. The solutions were then analyzed for conductivity, osmolarity, zeta potential, particle size distribution, viscosity, and surface tension following established methods [14,20,21].

2.8. Techno-Functional Properties of Citrus Pectin

The techno-functional properties of citrus peel powder were evaluated to assess its potential for food applications. Parameters including solubility, hygroscopicity, hydrophobicity, emulsifying capacity, foaming ability, and water- and oil-holding capacities were determined following established methods [14,20,21]. These measurements provide insight into the hydration, binding, and interfacial behavior of the material, which influence texture, stability, and overall performance in food and bioproduct systems.

2.9. Total Antioxidant Capacity of Citrus Pectin

Before the experiments, a citrus pectin solution (1.0% *w/v*) was prepared by dissolving 1.0 g of pectin powder in 100 mL of distilled water. The pH of the solution was then adjusted to 8.0 using ethanolamine to ensure complete solubilization of the pectin. The solution was incubated at 40 °C for 24 h. Subsequently, insoluble large particles were removed by centrifugation at 3000× *g* for 10 min. The resulting supernatant was collected and used for antioxidant analysis. Total antioxidant capacity was determined by mixing 20 μL of the sample solution with 1000 μL of 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) radical solution, followed by measurement of absorbance at 734 nm. A calibration curve was constructed using Trolox as the standard reference. Antioxidant activity was expressed as milligrams of Trolox equivalents (TE) per gram of sample. The percentage of radical scavenging activity and the half-maximal inhibitory concentration (IC₅₀) were calculated based on the change in absorbance at 734 nm [22].

2.10. Amylase Inhibitory Activity of Citrus Pectin

The α -amylase inhibitory activity was evaluated according to a previously established method with slight modifications, as described in earlier studies [20]. Briefly, 20 μ L of α -amylase solution (5.0 U/mL) was pre-incubated with either 30 μ L of pectin extract or acarbose (0–1000 μ g/mL) at 37 °C for 20 min. Subsequently, 250 μ L of soluble starch solution (10 mg/mL) prepared in 100 mM phosphate buffer (pH 7.0) was added to the reaction mixture, followed by incubation at 37 °C for an additional 30 min. The reaction was terminated by adding 20 μ L of 1.0 M HCl. To quantify the remaining undigested starch, 100 μ L of iodine reagent was added to each sample. Distilled water (2.0 mL) was then added to all tubes, and the absorbance was measured at 580 nm. A calibration curve was generated using acarbose as the positive control for α -amylase inhibition. The percentage inhibition of α -amylase activity and the half-maximal inhibitory concentration (IC₅₀) were calculated based on the changes in absorbance.

2.11. Statistical Analysis

The data were analyzed using SPSS Statistics for Windows, Version 21.0 (SPSS Inc., Chicago, IL, USA). Prior to statistical analysis, data normality and homogeneity of variances were assessed. Differences among treatments were evaluated using one-way analysis of variance (ANOVA). When significant differences were detected, Tukey's post hoc test was applied for multiple comparisons. Statistical significance was defined at $p \leq 0.05$, and results were expressed as mean $\pm$ standard deviation (Mean $\pm$ SD).

3. Results and Discussion

3.1. Biochemical Composition of Citrus Pectin

The biochemical composition of citrus peel pectin showed clear species-dependent variation when compared with commercial standard pectin (Table 1). The standard pectin contained 56.5 g/100 g dw pectin. In comparison, citrus species exhibited pectin contents ranging from 51.99 g/100 g dw in *C. maxima* to 55.49 g/100 g dw in *C. limon*, indicating that several citrus peels, particularly lemon, grapefruit, sweet orange, and mandarin, represent competitive natural sources of industrial pectin. Cellulose content (17.60–19.50 g/100 g dw) and hemicellulose content (15.30–16.20 g/100 g dw) were slightly higher in citrus peels than in the standard pectin sample, reflecting the more complex and less processed structure of plant cell wall materials. Similarly, lignin content ranged from 8.65 to 9.60 g/100 g dw, exceeding that of the commercial reference (8.36 g/100 g dw), which is consistent with the structural and protective roles of citrus peel cell walls. Structural protein content remained low across all samples (2.56–3.00 g/100 g dw) and was slightly lower than in standard pectin (3.74 g/100 g dw), likely due to differences in purification intensity. From a physiological perspective, pectin contributes to cell adhesion, firmness, water retention, and overall tissue integrity, while cellulose provides mechanical strength and structural support. Hemicellulose interacts with cellulose and pectin to enhance flexibility and cohesion of the cell wall, and lignin increases mechanical resistance and protection against microbial and oxidative stress. Structural proteins, although present in minor amounts, contribute to the stabilization of the polysaccharide network and may influence pectin extraction behavior [23].

Table 1. The biochemical composition of citrus pectin.

Biochemical Composition	<i>Citrus aurantium</i>	<i>Citrus limetta</i>	<i>Citrus limon</i>	<i>Citrus maxima</i>	<i>Citrus paradisi</i>	<i>Citrus reticulata</i>	<i>Citrus sinensis</i>	Sigma Pectin
Pectin (g/100 g)	52.74 $\pm$ 2.7 ^a	53.32 $\pm$ 2.5 ^a	55.49 $\pm$ 2.8 ^a	51.99 $\pm$ 2.5 ^a	54.82 $\pm$ 3.0 ^a	53.49 $\pm$ 2.6 ^a	54.40 $\pm$ 3.0 ^a	56.5 $\pm$ 3.0 ^a
Cellulose (g/100 g)	19.50 $\pm$ 1.6 ^a	18.17 $\pm$ 1.1 ^a	17.70 $\pm$ 1.0 ^a	19.00 $\pm$ 1.5 ^a	17.60 $\pm$ 1.4 ^a	18.00 $\pm$ 1.4 ^a	17.60 $\pm$ 1.3 ^a	16.50 $\pm$ 1.5 ^a

Table 1. Cont.

Biochemical Composition	<i>Citrus aurantium</i>	<i>Citrus limetta</i>	<i>Citrus limon</i>	<i>Citrus maxima</i>	<i>Citrus paradisi</i>	<i>Citrus reticulata</i>	<i>Citrus sinensis</i>	Sigma Pectin
Hemicellulose (g/100 g)	15.30 ± 1.3 ^a	16.00 ± 1.2 ^a	15.40 ± 1.2 ^a	16.20 ± 1.3 ^a	15.50 ± 1.5 ^a	15.80 ± 1.3 ^a	16.10 ± 1.3 ^a	14.37 ± 1.2 ^a
Lignin (g/100 g)	9.33 ± 0.8 ^a	9.60 ± 0.7 ^a	8.75 ± 0.6 ^a	9.20 ± 0.8 ^a	9.30 ± 0.7 ^a	9.27 ± 0.6 ^a	8.65 ± 0.2 ^a	8.36 ± 0.2 ^a
Protein (g/100 g)	2.77 ± 0.3 ^a	2.80 ± 0.2 ^a	2.56 ± 0.2 ^a	2.80 ± 0.3 ^a	2.65 ± 0.3 ^a	3.00 ± 0.4 ^a	2.88 ± 0.3 ^a	3.74 ± 0.4 ^a
Degree of methyl esterification (%)	52.07 ± 3.0 ^a	57.68 ± 3.2 ^a	35.16 ± 2.5 ^c	57.20 ± 2.4 ^a	45.28 ± 2.2 ^b	52.96 ± 3.0 ^a	45.04 ± 2.5 ^b	45.03 ± 2.3 ^b

Values are expressed as mean ± standard deviation (SD) of three replicates. Means followed by different letters within the same row are significantly different ($p < 0.05$) according to Tukey's test.

3.2. FTIR Spectroscopic Analysis and DM Determination of Citrus Pectin

FTIR spectroscopy was employed to characterize the structural features and functional groups of citrus peel pectins, along with associated biopolymers such as cellulose, hemicellulose, lignin, and structural proteins. The spectra of all extracted pectins exhibited highly similar profiles to commercial pectin (Figure 1), confirming the presence of galacturonic acid-rich polysaccharides. Minor spectral differences among citrus species were observed, likely reflecting variations in the degree of methyl esterification, intermolecular interactions, and compositional heterogeneity. A broad absorption band in the region of 3350–3290 cm^{-1} was assigned to O–H stretching vibrations associated with hydroxyl groups and extensive intra- and intermolecular hydrogen bonding within polysaccharide chains (Table 2). The weak band at 2950–2900 cm^{-1} corresponds to C–H stretching vibrations of aliphatic CH_2 and CH_3 groups. The absorption band observed at 1740–1730 cm^{-1} is characteristic of C=O stretching of esterified carboxyl groups ($-\text{COOCH}_3$), indicating the presence of methyl-esterified galacturonic acid residues and providing qualitative evidence of the degree of esterification. In contrast, the band at 1650–1600 cm^{-1} is attributed to asymmetric stretching of ionized carboxylate groups (COO^-), with overlapping contributions from amide I vibrations of proteins and aromatic skeletal vibrations of lignin. The relative intensities of these bands suggest structural variation in esterification among the samples. The region between 1550 and 1450 cm^{-1} includes contributions from amide II bands (proteins), CH_2 deformation vibrations of polysaccharides, and aromatic skeletal vibrations from lignin. A weak band around 1410 cm^{-1} , assigned to symmetric stretching of COO^- groups, indicates the presence of de-esterified galacturonic acid units, consistent with partially esterified pectin structures. The 1300–1000 cm^{-1} region represents the fingerprint of polysaccharides and is dominated by vibrations associated with the pectin backbone. Characteristic peaks at approximately 1330–1320 cm^{-1} (pyranose ring vibrations), 1230–1210 cm^{-1} (C–O stretching and side-chain vibrations), 1160–1150 cm^{-1} (C–O–C asymmetric stretching), 1110–1100 cm^{-1} (glycosidic linkages), and 1075–1015 cm^{-1} (C–O and C–C stretching) confirm the presence of α -(1→4)-linked galacturonic acid units (Table 2). Additional signals indicate the coexistence of other structural polysaccharides. Cellulose is evidenced by bands at 1160–1155, 1105, 1055, and 1030 cm^{-1} , along with a diagnostic β -glycosidic linkage at $\sim 898 \text{ cm}^{-1}$. Hemicellulose contributions are suggested by absorptions near 1730 cm^{-1} (acetyl/uronic esters) and 1245–1230 cm^{-1} (arabinoxylan linkages). Lignin is identified by aromatic skeletal vibrations at 1600–1595 cm^{-1} and 1515–1505 cm^{-1} , as well as methoxy group vibrations near 1270 cm^{-1} . Protein-related bands are observed at 1650–1640 cm^{-1} (amide I), 1540–1530 cm^{-1} (amide II), and 1270–1240 cm^{-1} (amide III). In the lower fingerprint region (1000–400 cm^{-1}), bands corresponding to glycosidic linkages and ring vibrations were observed. Signals near 978 cm^{-1} are associated with β -glucan linkages, while bands around 840–830 cm^{-1} correspond to α -glycosidic configurations. Additional peaks in the 750–600 cm^{-1} range are attributed to skeletal vibrations of C–C and

C–O bonds. These features confirm the coexistence of both α - and β -linked polysaccharide structures, contributing to the functional and nutritional properties of citrus-derived fibers.

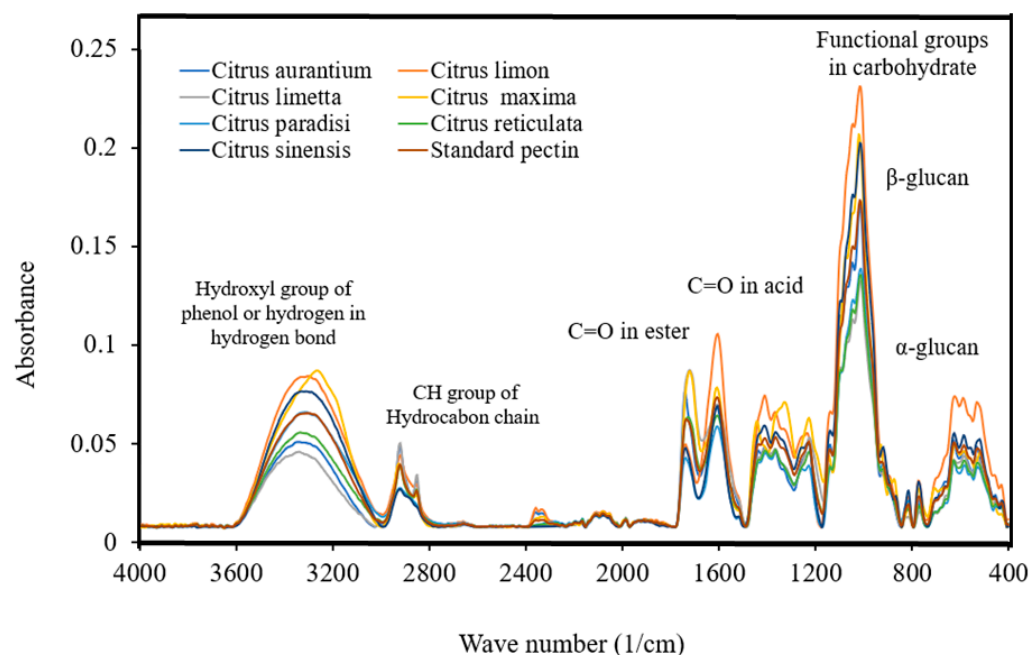


Figure 1. FTIR spectra of pectin extracted from seven citrus species compared with commercial pectin. Characteristic absorption bands correspond to O–H stretching ($3350\text{--}3290\text{ cm}^{-1}$), aliphatic C–H stretching ($2950\text{--}2900\text{ cm}^{-1}$), esterified/ionized carboxyl C=O stretching (~ 1730 and $1600\text{--}1650\text{ cm}^{-1}$). The carbohydrate fingerprint region ($1200\text{--}1000\text{ cm}^{-1}$), associated with C–O–C and glycosidic bond vibrations, is prominent. Signals corresponding to α - and β -glycosidic linkages (~ 830 and $\sim 890\text{ cm}^{-1}$) are also observed.

Table 2. FTIR peak assignments of citrus peel pectins and associated biopolymers (cellulose, hemicellulose, lignin, and proteins).

Wavenumber (cm^{-1})	Functional Group/Vibration	Pectin	Cellulose	Hemicellulose	Lignin	Protein
3350–3290	O–H stretching, hydrogen bonding	+	+	+	–	–
2950–2900	C–H stretching (CH_2 , CH_3)	+	+	+	weak	–
2350	N–H stretching of amidated groups	+ (low)	–	–	–	–
1740–1730	C=O stretching of esterified COOH ($-\text{COOCH}_3$)	+ (degree of esterification)	–	+ (acetyl/uronic/esters)	–	–
1650–1600	Ionized carboxylate (COO^-), amide I, aromatic C=C	+ (COO^-)	–	–	+ (aromatic skeletal)	+ (amide I)
1550–1530	Amide II, N–H bending	–	–	–	–	+ (amide II)
1515–1505	Aromatic skeletal vibrations	–	–	–	+	–
1460–1450	CH_2 deformation	+	+	+	+	–
1410	Symmetric COO^- stretching	+	–	–	–	–
1370–1360	C–H bending (CH , CH_3)	+	+	+	–	–

Table 2. Cont.

Wavenumber (cm ⁻¹)	Functional Group/Vibration	Pectin	Cellulose	Hemicellulose	Lignin	Protein
1330–1320	Pyranose ring vibration	+	–	–	–	–
1270–1240	C–O stretch (methoxy groups), amide III	–	–	+ (arabinoxylan)	+ (guaia-cyl/syringyl CH–O)	+ (amide III)
1230–1210	Side-chain C–O–C vibrations	+	–	+	–	–
1160–1150	C–O–C asymmetric stretching	+	+ (cellulose I band)	+	+	–
1110–1100	Ring vibration, glycosidic stretching	+	+	+	+	–
1075–1030	C–O, C–C stretching of polysaccharide backbone	+	+	+	weak	–
1015	C–O stretching of galacturonic acid	+	–	–	–	–
978	β-glucan, β-1,3/β-1,4 linkages	–	+	+	–	–
897–890	β-glycosidic linkage	+ (weak)	+ (strong cellulose marker)	+	–	–
840–830	α-glycosidic linkages	+	–	–	–	–
830–750	Aromatic C–H out-of-plane bending	–	–	–	+	–
750–600	Skeletal C–C, C–O–C bending	+	+	+	+	–
600–500	Fingerprint region of polysaccharides	+	+	+	–	–

Overall, the FTIR spectra confirm that citrus pectins consist predominantly of galacturonic acid-based polysaccharides with varying degrees of methyl esterification. The presence of cellulose, hemicellulose, lignin, and minor protein components highlights the structural complexity of citrus peel biomass. The relative intensities of ester ($\sim 1730\text{ cm}^{-1}$) and carboxylate ($\sim 1600\text{ cm}^{-1}$) bands further suggest variability in esterification among samples, which may influence their physicochemical and gelling properties [24,25]. The relative intensities of the characteristic absorbance bands were used to quantify the degree of methyl esterification (DM) for each sample, as presented in Table 1. A marked interspecific variation was observed in the DM, ranging from 35.16% in lemon to 57.68% in *C. limetta*. This parameter is functionally critical because it determines pectin's gelation behavior and suitability for different applications. High-methoxyl pectins (DM > 50%), such as those from *C. limetta*, *C. maxima*, and *C. reticulata*, are suitable for high-sugar, low-pH systems and are widely used in jams, jellies, fruit preserves, and confectionery products. In contrast, low-methoxyl pectins (DM < 50%), such as those from lemon (35.16%) and grapefruit (45.28%), form gels in the presence of divalent cations such as Ca^{2+} and are preferred for reduced-sugar formulations, yogurt and acidified dairy gels, beverages, and dietary fiber-enriched functional foods. Overall, the observed biochemical diversity highlights the potential of citrus peel-derived pectins as versatile functional ingredients. Lemon,

grapefruit, sweet orange, and mandarin peels, in particular, represent valuable sources of pectin with properties comparable to or even exceeding those of commercial standards [26]. Their varying DM values enable a broad range of technological applications, including gelling, stabilizing, thickening, emulsifying, and dietary fiber enrichment, with associated health benefits. The structural characteristics of citrus pectins also confer resistance to hydrolysis under simulated gastrointestinal conditions, supporting their use in low-calorie and anti-diabetic formulations by potentially modulating glucose release and improving glycemic control. Furthermore, citrus peel polysaccharides may act as prebiotics, promoting the growth of beneficial gut microbiota and enhancing the functional value of food products [27].

3.3. Ultraviolet Light Absorption of Citrus Pectin

UV-Vis spectroscopy is widely used in food, nutraceutical, and beverage analysis to assess sample purity, detect co-extracted compounds, and evaluate the integrity of natural biopolymers. The UV absorbance spectra of citrus pectin are presented in Figure 2. Citrus pectin solutions showed a prominent absorbance peak around 220 nm, corresponding to electronic transitions of carbonyl (C=O) and carboxyl (–COOH) groups within the galacturonic acid backbone. This band is a typical feature of pectin and reflects the presence of both esterified and free carboxylic groups [18]. A secondary absorbance region between 250 and 290 nm was also observed across all samples, with varying intensity depending on the species. This band is attributed to aromatic and conjugated compounds, including phenolic substances, lignin-derived residues, and trace proteins. These components are commonly co-extracted during pectin isolation due to their strong association with the plant cell-wall matrix, and their presence is consistent with UV profiles of plant-derived pectins rather than indicating contamination; instead, it reflects the inherent biochemical complexity of citrus peel biomass [18]. Cellulose and hemicellulose, as non-aromatic polysaccharides, exhibited minimal absorbance above 240 nm, typically showing only a weak shoulder near 200–210 nm due to C–O and C–H electronic transitions. In contrast, lignin displayed broad absorbance across both the 200–240 nm and 260–300 nm regions, consistent with its highly aromatic phenylpropanoid structure and conjugated systems. Structural proteins showed strong absorbance in the 190–230 nm region, corresponding to peptide bond transitions, along with a weaker band near 280 nm associated with aromatic amino acids such as tryptophan, tyrosine, and phenylalanine [28]. Overall, the UV-Vis absorbance profiles indicate that citrus pectin is structurally comparable to standard commercial pectin, while the presence of minor amounts of lignin, phenolics, and proteins reflects the natural biochemical complexity of citrus peel cell-wall materials. These spectral characteristics are important for evaluating extraction purity, optimizing processing conditions, and predicting functional performance in food systems, particularly in relation to optical clarity, color stability, and antioxidant properties [28].

3.4. Fluorescence Emission of Citrus Pectin

Fluorescence spectroscopy is a sensitive analytical technique widely applied in food and beverage quality control for detecting trace contaminants, structural modifications, and biomolecular interactions in complex matrices [29]. The fluorescence emission profiles of citrus pectin are presented in Figure 3. Standard commercial pectin exhibited a characteristic emission maximum at 360 nm upon excitation at 280 nm, which is typically associated with intrinsic fluorescence arising from trace aromatic residues or minor protein impurities. In contrast, pectin extracted from *Citrus aurantium* and *Citrus maxima* showed a shifted and more intense emission peak at approximately 420 nm, suggesting the presence of co-extracted chromophoric compounds with higher degrees of conjugation or aromaticity.

Pectins derived from *C. limetta*, *C. limon*, *C. paradisi*, *C. reticulata*, and *C. sinensis* displayed broader emission bands spanning 420–500 nm, with a maximum around 460 nm, indicating greater heterogeneity in associated secondary metabolites. All citrus pectin samples also exhibited a consistent secondary emission peak near 560 nm, which may be attributed to carotenoid-like compounds or oxidized phenolics retained during extraction. These molecules possess extended π -conjugated systems that enable fluorescence emission in the visible region [29]. The interspecific differences observed among citrus peels reflect their inherent biochemical diversity. Although cellulose and hemicellulose are not intrinsically fluorescent, lignin and structural proteins contribute significantly to the observed emission profiles. Lignin exhibits broad fluorescence in the 350–500 nm range due to its phenylpropanoid structure, while structural proteins containing aromatic amino acids (tryptophan, tyrosine, and phenylalanine) typically fluoresce strongly between 330 and 365 nm. The co-extraction of these components with pectin introduces additional fluorescent signals that influence the overall emission behavior [30]. Overall, the observed spectral shifts, emission broadening, and intensity variations among citrus-derived pectins indicate the presence of aromatic amino acids, polyphenols, and lignin-derived residues interacting with the pectin matrix. These interactions may modulate fluorescence through quenching, energy transfer, or enhancement mechanisms. Consequently, fluorescence spectroscopy provides valuable information regarding the purity, structural composition, and functional quality of citrus pectins for food-related applications [31].

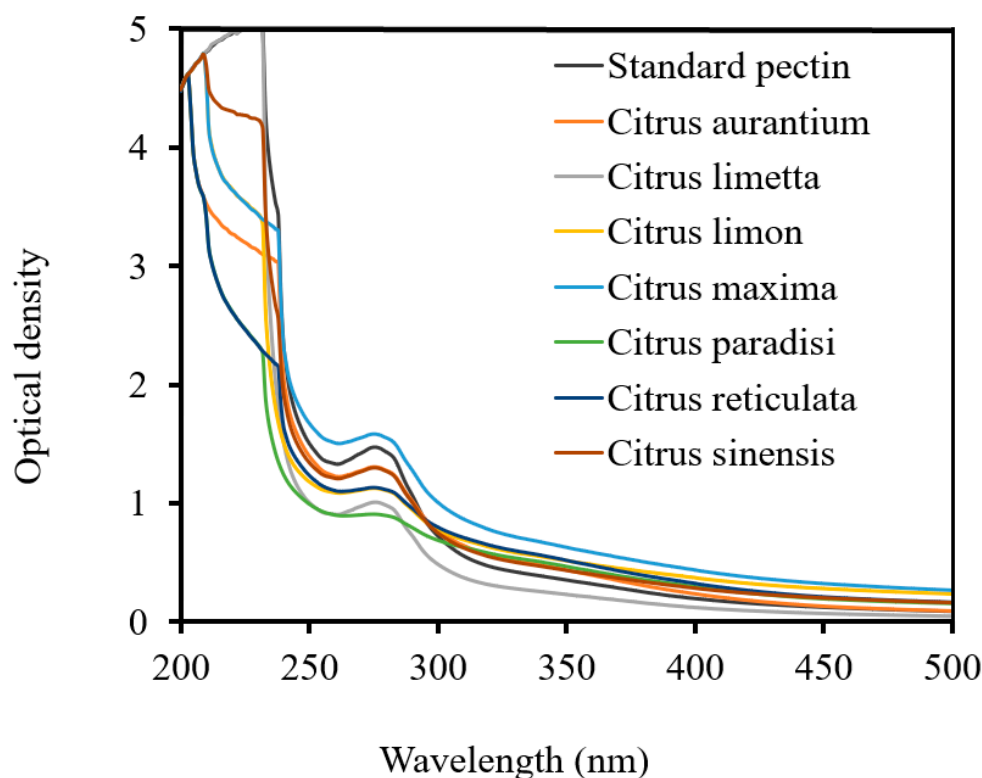


Figure 2. UV-Vis absorbance spectra of citrus pectin solutions compared with standard pectin. The spectra exhibit a strong absorption peak around 220 nm, attributed to carbonyl (C=O) and carboxyl (–COOH) groups in the pectin backbone. A secondary absorption band in the range of 250–290 nm is also observed, which may be associated with aromatic residues, proteins, or phenolic compounds co-extracted during the pectin isolation process.

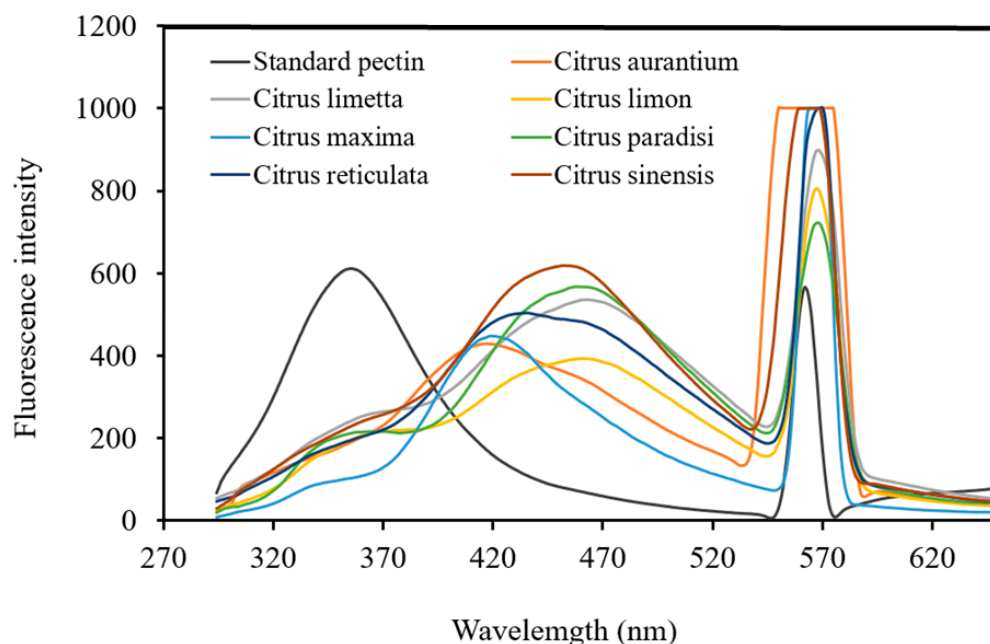


Figure 3. Fluorescence emission spectra of citrus pectin solutions compared with standard pectin. Upon excitation at 280 nm, standard pectin exhibits an emission maximum at 360 nm. In contrast, pectins extracted from *Citrus aurantium* and *Citrus maxima* show a shifted emission around 420 nm. Other citrus-derived pectins display broader emission in the 420–500 nm range, with a maximum near 469 nm. Additionally, all citrus pectin samples exhibit a secondary narrow emission peak at approximately 560 nm.

3.5. Thermal Analysis of Citrus Pectin: TGA, DTG, and DSC

The thermal behavior of citrus-derived pectin was evaluated using TGA, DTG, and DSC, providing complementary insights into mass loss, degradation kinetics, and thermal transitions of the biopolymer matrix (Figure 4, Figure 5 and Figures S1–S16). The combined profiles reveal a characteristic multistep degradation pattern reflecting the complex composition of citrus pectin, which includes not only galacturonic acid-based polysaccharides but also associated hemicellulose, cellulose, lignin, and residual proteins.

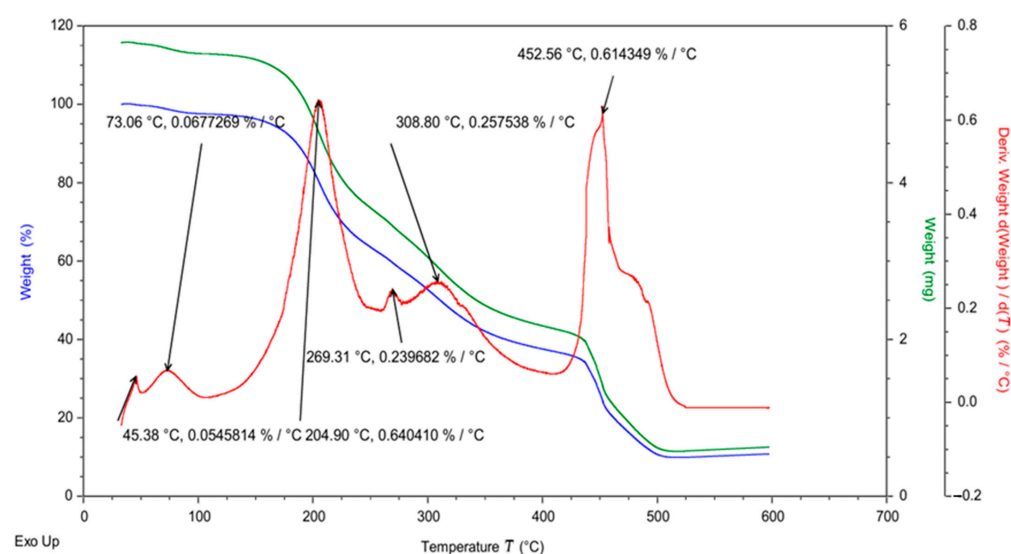


Figure 4. TG and DTG profiles of *Citrus sinensis* pectin showing multi-stage thermal degradation. Data for all other isolated citrus pectin samples are provided in the Supplementary Materials (Figures S1–S8).

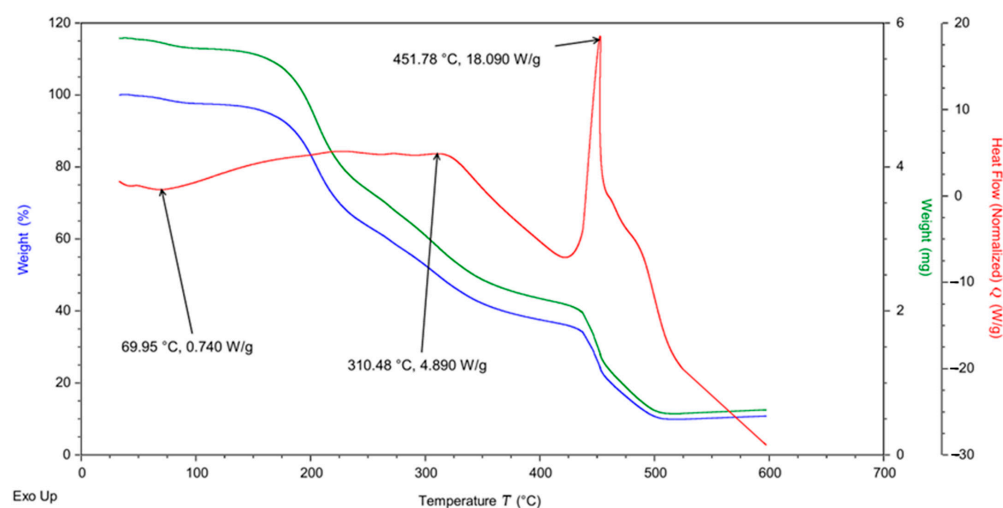


Figure 5. Combined TGA and DSC curves of *Citrus sinensis* pectin demonstrate distinct endothermic and exothermic thermal transitions. Data for all other isolated citrus pectin samples are provided in the Supplementary Materials (Figures S9–S16).

The TGA-DTG curves demonstrate sequential weight-loss events corresponding to moisture removal and progressive thermal decomposition of structural components. An initial minor mass loss occurs around 70 °C, attributed to the evaporation of free and weakly bound water, as supported by a low-intensity DTG signal. The second stage, centered near 230–240 °C, represents the onset of pectin degradation, involving cleavage of esterified galacturonic acid chains and partial decomposition of hemicellulose-like fractions. A more pronounced degradation step around 280–290 °C corresponds to further depolymerization of polysaccharides and breakdown of side-chain substituents, including contributions from thermally sensitive proteins.

A subsequent transition near 340–350 °C is associated with decomposition of more ordered carbohydrate domains and the initial degradation of cellulose microstructures, indicating reduced stability of semi-crystalline regions. The most intense degradation event occurs at approximately 450–470 °C, characterized by a sharp DTG peak and rapid mass loss, corresponding to the breakdown of residual cellulose and oxidative degradation of lignin-rich fractions, leading to char formation. Above 500 °C, minimal mass changes are observed, indicating the presence of thermally stable carbonaceous residues and ash. This multistep pattern confirms the heterogeneous and lignocellulosic nature of citrus-derived pectin [32].

Comparative analysis across citrus species shows similar degradation profiles, with slight variations in peak temperatures due to differences in degree of esterification (DE), purity, and residual composition. High-methoxyl pectins tend to exhibit slightly shifted degradation temperatures due to stronger hydrophobic interactions, whereas impurities such as proteins, ash, and cellulose introduce additional minor transitions. Consistent with the literature, pectin typically degrades at lower temperatures (150–220 °C), followed by hemicellulose (220–310 °C), cellulose (330–370 °C), and finally lignin, which decomposes gradually over a wide range, with a major contribution at 450–500 °C [33–35].

The DSC thermograms further support the multistep thermal behavior observed in TGA. Prior to major transitions, a subtle baseline shift may indicate a glass transition (T_g) associated with amorphous pectin domains, reflecting increased molecular mobility influenced by moisture content and interactions with co-existing biopolymers. The first distinct endothermic peak appears at approximately 60–65 °C, corresponding to the removal of physically bound water and relaxation of weakly associated polysaccharide-protein networks. A broad exothermic transition centered around 310–320 °C represents the primary

decomposition stage, involving depolymerization of pectin chains, cleavage of glycosidic linkages in cellulose and hemicellulose, and partial degradation of proteinaceous components. This region may also include structural rearrangements or polymorphic transitions in semi-crystalline domains, particularly cellulose. The degree of esterification significantly influences this transition, as higher DE reduces hydrogen bonding and may lower thermal stability [34,36].

The most prominent DSC feature is a sharp exothermic peak at approximately 450–460 °C, corresponding to oxidative degradation of carbonaceous residues and lignin-like structures. This event reflects combustion-like processes rather than true melting, as pectin and related polysaccharides do not exhibit a conventional melting point but instead undergo thermal decomposition at elevated temperatures. The persistence of thermal events beyond 400 °C indicates the presence of lignin and other resistant components, particularly in less purified (crude) pectin samples.

Overall, the combined TGA-DTG-DSC analysis demonstrates that citrus pectin undergoes a complex, multi-stage thermal degradation process involving initial dehydration, possible glass transition-related softening, progressive polysaccharide and protein decomposition, and final oxidative breakdown of lignocellulosic residues. These findings are critical for optimizing thermal processing conditions, such as drying, extrusion, and film formation, and for designing thermally stable pectin-based materials. The degree of methoxylation and the presence of associated biopolymers play key roles in determining the thermal stability and functional performance of citrus-derived pectin [35,37].

3.6. XRD Analysis of Citrus Pectin

XRD analysis revealed that citrus pectin possesses a predominantly amorphous polymeric structure with limited short-range molecular ordering. The diffraction pattern displayed a broad halo in the range of $2\theta \approx 14\text{--}26^\circ$, with maximum intensity centered around $20\text{--}22^\circ$, indicating the absence of long-range crystalline organization (Figure 6). Using Cu K α radiation ($\lambda = 0.15406$ nm) and applying Bragg's law, the corresponding interplanar spacing (d-spacing) was estimated to fall within 0.40–0.63 nm. At the main scattering position ($2\theta \approx 20^\circ$), the calculated spacing (~ 0.44 nm) is consistent with short-range interactions among galacturonic acid chains and the irregular distribution of side groups typical of low-crystallinity pectin structures [38].

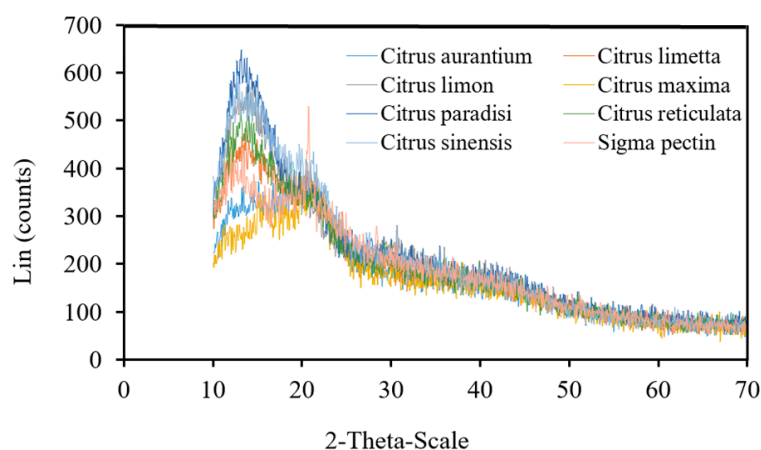


Figure 6. XRD pattern of Citrus aurantium pectin containing residual cellulose, hemicellulose, lignin, and structural proteins. The diffraction profile exhibits a broad amorphous halo in the range of $2\theta \approx 14\text{--}26^\circ$, with a maximum near 20° , indicating the predominantly amorphous nature of the sample. A weak shoulder around 22° is attributed to disordered cellulose microdomains, while the diffuse background reflects contributions from amorphous hemicellulose, lignin, and protein residues.

The broadness of the diffraction halo and the absence of sharp Bragg peaks confirm that the material lacks long-range periodicity and is dominated by amorphous organization. A weak shoulder observed near 22° ($d \approx 0.40$ nm) suggests the presence of highly disordered cellulose domains. However, the lack of characteristic reflections corresponding to cellulose I or cellulose II indicates that any residual cellulose is structurally disrupted and exists in a paracrystalline or nano-amorphous state. This behavior is consistent with partial depolymerization and structural distortion of cellulose microfibrils during pectin extraction, resulting in reduced crystallinity and overlap with the main amorphous halo [39]. Hemicellulose and lignin, which are intrinsically amorphous, contribute primarily to the diffuse background scattering. Their presence broadens the overall peak profile and increases the full width at half maximum (FWHM), further masking any weak crystalline features. In particular, lignin, with its complex cross-linked aromatic structure, introduces significant structural heterogeneity, contributing to the gradual decay of scattering intensity beyond 30° . This behavior aligns with disorder-based scattering models, such as the Debye scattering function, typically observed in non-periodic macromolecular systems [40]. Overall, the XRD profile confirms that citrus pectin, along with its associated biopolymers (cellulose, hemicellulose, lignin, and structural proteins), forms a structurally heterogeneous system dominated by amorphous characteristics, short-range molecular ordering, and limited coherence length. The absence of distinct crystalline reflections indicates that the material can be described by a paracrystalline disorder model, with significant deviations from ideal lattice structure. This predominantly amorphous architecture is consistent with the functional behavior of pectin in food systems, where flexibility, hydration capacity, and gel-forming ability are closely linked to its non-crystalline nature [41].

3.7. Physicochemical and Rheological Properties of Citrus Pectin Solutions

Citrus pectin solutions exhibited negative zeta potential, nanoscale particle size, and relatively high conductivity, osmolarity, and surface activity, as summarized in Table 3. These physicochemical properties showed clear interspecific variability, primarily driven by differences in DM, molecular aggregation, and the presence of neutral sugars and co-extracted biopolymers. Such variations directly influence colloidal stability and techno-functional performance, particularly in food systems where pectin is used as a thickening, stabilizing, and gelling agent [42]. Rheological analysis revealed notable differences among citrus species. Shear stress values ranged from 1982 mPa (*C. aurantium*) to 3273 mPa (*C. maxima*), indicating stronger network formation in samples with higher DM, osmolarity, and smaller particle size. Viscosity followed a similar trend (7.96–13.15 mPa·s), with *C. maxima* exhibiting the highest thickening capacity. All pectin solutions (1%) demonstrated typical non-Newtonian shear-thinning (pseudoplastic) behavior, as shown by the decrease in viscosity with increasing shear rate (Figure 7), which is a common feature of high-molecular-weight polysaccharides. This behavior arises from the alignment of pectin chains under shear, which reduces intermolecular interactions and flow resistance. The extent of shear-thinning is influenced by structural parameters such as molecular weight, degree of esterification, and branching (e.g., rhamnogalacturonan regions), although all samples in this study exhibited comparable flow patterns at the tested concentration [43]. These properties are advantageous in food applications, allowing easy flow under shear (e.g., pouring) while maintaining viscosity and stability at rest. The DM ranged from 35.16% (*C. limon*) to 57.68% (*C. limetta*), classifying the samples into low- and high-methoxyl pectins. High-DM pectins (*C. maxima*, *C. limetta*, *C. reticulata*) favor hydrophobic interactions and sugar-dependent gelation, whereas low-DM pectins (*C. limon*, *C. paradisi*, *C. sinensis*) are more suitable for calcium-mediated gel formation [42]. Electrical conductivity varied moderately (720–750 $\mu\text{S}/\text{cm}$), with *C. maxima* and commercial pectin showing

higher values, likely due to increased levels of soluble ions and low-molecular-weight components. Surface tension ranged from 52 to 65 mN/m, with lower values (e.g., *C. limetta* and commercial pectin) indicating greater surface activity, possibly linked to residual proteins or neutral sugars, while higher values (e.g., *C. reticulata*, *C. paradisi*) suggest weaker interfacial properties [44]. Osmolarity values (74–90 mOsm/kg) reflected solute concentration, with *C. maxima* showing the highest osmotic pressure, consistent with a more soluble or less branched structure. Increased osmolarity enhances particle interactions, contributing to higher viscosity and shear resistance. Similarly, stronger intermolecular interactions and higher surface tension can further increase viscosity by reinforcing particle–particle associations [44]. All citrus pectin samples exhibited strongly negative zeta potential values (−55 to −58 mV), indicating excellent colloidal stability due to strong electrostatic repulsion. In contrast, commercial pectin (−41 mV) showed comparatively lower stability, suggesting that citrus-derived pectins may offer superior dispersion performance. High zeta potential increases hydrodynamic volume and particle mobility, contributing to enhanced viscosity, whereas lower values may promote aggregation and eventual sedimentation [45]. Particle size ranged from 137 to 156 nm, with *C. maxima* showing the smallest particles, which likely enhances hydration and viscosity. Larger particles observed in *C. limon* and commercial pectin were associated with weaker network formation. The polydispersity index (PDI: 0.26–0.37) indicated generally monodisperse systems, although *C. sinensis* showed a slightly broader size distribution. Smaller particles, due to their higher surface area and increased Brownian motion, contribute to higher viscosity, while polydispersity can influence packing efficiency and flow behavior [46]. Overall, *C. maxima* demonstrated superior physicochemical and rheological properties, making it a promising candidate for applications requiring high viscosity, strong gelation, and stable colloidal systems. In contrast, *C. limon* and *C. sinensis*, with lower DM and moderate viscosity, are better suited for calcium-induced gel systems and reduced-sugar formulations. These findings indicate that citrus-derived pectins are not only comparable to commercial pectin but, in several aspects, may offer enhanced stability and functional performance [47].

Table 3. Physico-chemical properties of citrus pectin.

Physicochemical Properties	<i>Citrus aurantium</i>	<i>Citrus limetta</i>	<i>Citrus limon</i>	<i>Citrus maxima</i>	<i>Citrus paradisi</i>	<i>Citrus reticulata</i>	<i>Citrus sinensis</i>	Sigma Pectin
Conductivity (μS/cm)	720.00 ± 18 ^a	735.00 ± 15 ^a	740.00 ± 21 ^a	750.00 ± 20 ^a	744.00 ± 23 ^a	737.00 ± 19 ^a	730.00 ± 18 ^a	750.00 ± 25 ^a
Surface tension (mN/m)	54.00 ± 2.80 ^a	52.00 ± 2.65 ^a	55.00 ± 2.50 ^a	57.00 ± 3.00 ^a	62.00 ± 3.80 ^a	65.00 ± 3.60 ^a	58.00 ± 3.30 ^a	52.00 ± 3.75 ^a
Interfacial tension (mN/m)	20.00 ± 1.77 ^a	17.00 ± 1.50 ^{bc}	18.00 ± 1.40 ^{ab}	21.00 ± 1.80 ^a	15.00 ± 1.33 ^c	19.00 ± 2.0 ^a	22.00 ± 2.20 ^a	21.00 ± 1.86 ^a
Osmolarity (mOsmol/kg)	77.00 ± 3.55 ^{bc}	80.00 ± 4.18 ^b	86.00 ± 4.30 ^a	90.00 ± 4.80 ^a	74.00 ± 3.77 ^c	85.00 ± 4.32 ^a	78.00 ± 3.90 ^{bc}	74.00 ± 4.00 ^c
Zeta-potential (mV)	57.70 ± 2.80 ^a	56.30 ± 2.37 ^a	58.20 ± 2.50 ^a	58.30 ± 2.78 ^a	55.30 ± 3.00 ^a	58.40 ± 3.17 ^a	58.40 ± 2.88 ^a	41.00 ± 2.46 ^b
Particle size (nm)	148.0 ± 6.7 ^b	151.0 ± 7.0 ^a	156.0 ± 7.2 ^a	137.0 ± 5.7 ^c	145.0 ± 5.3 ^b	141.0 ± 5.0 ^c	154.0 ± 6.0 ^a	156.0 ± 5.0 ^a
Polydispersity	0.26 ± 0.01 ^a	0.32 ± 0.02 ^a	0.30 ± 0.02 ^a	0.34 ± 0.03 ^a	0.32 ± 0.02 ^a	0.30 ± 0.02 ^a	0.37 ± 0.03 ^a	0.30 ± 0.02 ^a
Viscosity (mPa.s)	7.96 ± 0.55 ^b	11.05 ± 0.93 ^a	11.49 ± 0.90 ^a	13.15 ± 1.00 ^a	9.99 ± 0.78 ^{ab}	8.10 ± 0.70 ^b	12.60 ± 1.10 ^a	11.22 ± 1.00 ^a

Values are expressed as mean ± standard deviation (SD) of three replicates. Means followed by different letters within the same row are significantly different ($p < 0.05$) according to Tukey's test.

3.8. Techno-Functional Properties of Citrus Pectin

Citrus pectins exhibited high swelling capacity, hygroscopicity, water-holding capacity (WHC), oil-holding capacity (OHC), and emulsifying activity, but relatively low foaming capacity (Table 4). These techno-functional properties are largely governed by the biochemical composition of the pectin, particularly the DM, as well as the presence of lignin, structural proteins, phenolics, and neutral sugar side chains [48]. Pectins with higher DM, such as *C. limetta* (57.68%) and *C. maxima* (57.20%), displayed moderate hydrophobicity

due to a lower proportion of free carboxyl groups and increased methyl esterification. This structural feature promotes partial hydrophobic interactions while maintaining sufficient hydration capacity [49]. In contrast, low-DM pectins, particularly *C. limon* (35.16%), exhibited higher water solubility (68–75%), greater hygroscopicity (~15.50%), and stronger water-binding capacity (~5.60 g/g), reflecting the abundance of ionized galacturonic acid groups capable of forming extensive hydrogen bonds [36]. Swelling behavior and WHC were also influenced by the presence of non-pectic polysaccharides such as cellulose and hemicellulose. For example, *C. maxima*, which contains higher levels of these structural carbohydrates, showed superior swelling (~49.00%) and WHC (~5.75 g/g), likely due to its more porous and fiber-rich matrix that enhances water retention [50]. Similarly, OHC is associated with the ability of hydrophobic domains within the pectin structure to interact with lipids, which is beneficial in fat-containing food systems.

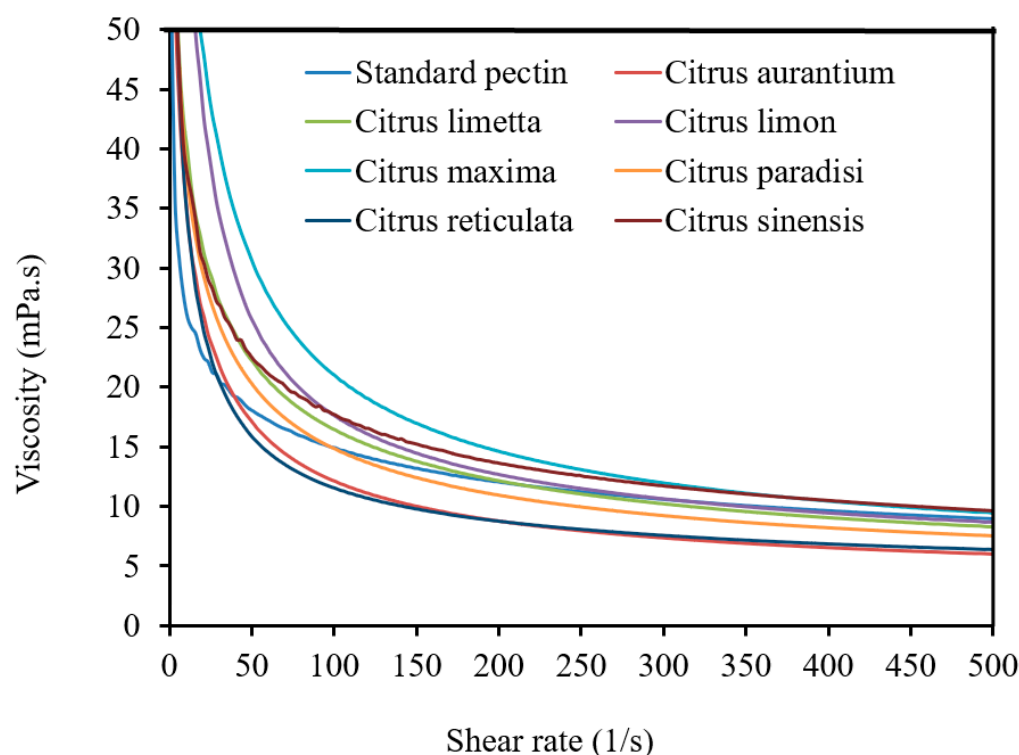


Figure 7. Viscosity-shear rate profiles of citrus pectin. All 1% pectin solutions exhibit non-Newtonian, shear-thinning (pseudoplastic) behavior.

Table 4. Techno-functional properties of citrus pectins.

Technofunctional Properties	<i>Citrus aurantium</i>	<i>Citrus limetta</i>	<i>Citrus limon</i>	<i>Citrus maxima</i>	<i>Citrus paradisi</i>	<i>Citrus reticulata</i>	<i>Citrus sinensis</i>	Sigma Pectin
Water swelling (%)	47.50 ± 2.5 ^a	52.30 ± 3.0 ^a	50.50 ± 2.8 ^a	49.00 ± 2.4 ^a	41.40 ± 2.3 ^b	48.00 ± 2.6 ^a	53.00 ± 3.2 ^a	53.00 ± 3.0 ^a
Water solubility (%)	72.00 ± 3.5 ^a	68.00 ± 3.7 ^{ab}	74.00 ± 4.0 ^a	75.00 ± 3.9 ^a	65.00 ± 4.2 ^b	67.00 ± 3.7 ^b	73.00 ± 4.2 ^a	75.00 ± 4.0 ^a
Water Hygroscopicity (%)	14.70 ± 1.20 ^{ab}	16.00 ± 1.5 ^a	15.50 ± 1.4 ^a	14.80 ± 1.3 ^{ab}	13.70 ± 1.2 ^b	14.00 ± 1.4 ^{ab}	15.00 ± 1.3 ^a	12.40 ± 1.0 ^b
Water hydrophobicity (μg/g)	0.85 ± 0.03 ^b	1.02 ± 0.06 ^a	0.93 ± 0.05 ^a	0.78 ± 0.04 ^b	0.90 ± 0.06 ^a	0.97 ± 0.05 ^a	0.86 ± 0.04 ^b	0.77 ± 0.04 ^c
Water holding capacity (g/g)	5.45 ± 0.25 ^a	5.17 ± 0.27 ^a	5.60 ± 0.23 ^a	5.75 ± 0.27 ^a	4.80 ± 0.22 ^{ab}	5.15 ± 0.20 ^a	4.70 ± 0.18 ^b	4.63 ± 0.17 ^b
Oil holding capacity (g/g)	2.50 ± 0.85 ^a	2.40 ± 0.77 ^a	2.35 ± 0.60 ^a	2.47 ± 0.70 ^a	2.33 ± 0.73 ^a	2.25 ± 0.67 ^a	2.40 ± 0.65 ^a	2.25 ± 0.73 ^a
Foaming capacity (%)	15.30 ± 1.70 ^a	16.50 ± 1.47 ^a	16.00 ± 1.35 ^a	15.70 ± 1.42 ^a	14.80 ± 1.25 ^a	14.50 ± 1.44 ^a	15.70 ± 1.37 ^a	14.50 ± 1.30 ^a
Foaming capacity (%)	12.20 ± 1.15 ^a	13.00 ± 1.22 ^a	11.00 ± 1.00 ^{ab}	12.00 ± 1.27 ^a	10.00 ± 1.00 ^b	9.00 ± 0.83 ^b	10.00 ± 0.90 ^b	12.30 ± 1.10 ^a
Foaming capacity (%)	8.00 ± 0.33 ^a	7.00 ± 0.28 ^a	5.00 ± 0.22 ^a	7.00 ± 0.40 ^a	6.00 ± 0.38 ^a	5.00 ± 0.26 ^a	6.00 ± 0.33 ^a	8.40 ± 0.40 ^a

Table 4. Cont.

Technofunctional Properties	<i>Citrus aurantium</i>	<i>Citrus limetta</i>	<i>Citrus limon</i>	<i>Citrus maxima</i>	<i>Citrus paradisi</i>	<i>Citrus reticulata</i>	<i>Citrus sinensis</i>	Sigma Pectin
Emulsifying activity (%)	75.00 ± 3.35 ^a	73.00 ± 3.80 ^a	70.00 ± 4.23 ^a	74.00 ± 4.50 ^a	67.00 ± 5.00 ^a	65.00 ± 4.90 ^a	70.00 ± 5.00 ^a	77.00 ± 5.20 ^a
Emulsion stability (%)	70.00 ± 3.45 ^a	68.00 ± 3.27 ^a	68.00 ± 3.50 ^a	70.00 ± 4.70 ^a	62.00 ± 4.00 ^a	63.00 ± 4.60 ^a	67.00 ± 4.70 ^a	72.00 ± 5.00 ^a
Emulsion stability (%)	67.00 ± 4.40 ^a	65.00 ± 4.63 ^a	64.00 ± 4.20 ^a	65.00 ± 4.00 ^a	60.00 ± 3.88 ^b	58.00 ± 3.50 ^b	63.00 ± 4.00 ^{ab}	69.00 ± 4.22 ^a

Values are expressed as mean ± standard deviation (SD) of three replicates. Means followed by different letters within the same row are significantly different ($p < 0.05$) according to Tukey's test.

Protein and phenolic residues significantly affected interfacial properties. Samples with relatively higher protein content, such as *C. sinensis* and *C. limetta*, exhibited slightly improved foaming capacity (15.7 and 16.5%) due to the surface-active nature of protein fractions. However, overall foaming capacity remained low across all samples. In contrast, pectins richer in phenolics or lignin (e.g., *C. paradisi*) showed reduced foam formation and stability, likely due to protein-phenolic interactions and steric hindrance at the air-water interface [50].

Emulsifying activity (65–75%) and emulsion stability (60–70%) were strongly influenced by both DM and associated biopolymers. High-DM pectins (e.g., *C. aurantium* and *C. maxima*) demonstrated stronger initial emulsification due to enhanced adsorption at oil-water interfaces. Additionally, neutral sugars such as arabinose and galactose, present in branched side chains, contributed to the steric stabilization of oil droplets, thereby improving long-term emulsion stability. Residual proteins, acetyl groups, and phenolic compounds can further enhance interfacial activity by reducing interfacial tension and strengthening the interfacial film [51,52]. Previous studies indicate that emulsifying performance varies significantly depending on pectin source and structure. Pectins with higher molecular weight, compact conformation, and higher esterification tend to form more stable emulsions due to improved viscosity and interfacial film formation. Structural modifications, such as glycosylation or controlled Maillard reactions, can further enhance emulsifying properties by improving protein-polysaccharide interactions and droplet stabilization [53]. Overall, the functional behavior of citrus pectins is determined by a combination of structural and compositional factors. Higher DM enhances hydrophobic interactions and interfacial adsorption, while neutral sugar side chains improve hydration, viscosity, and steric stabilization. Conversely, lignin and phenolic residues may reduce solubility or flexibility, and protein content can either enhance or interfere with functionality depending on its concentration. These combined attributes result in species-specific techno-functional profiles. Consequently, selecting appropriate citrus sources or optimizing extraction and processing conditions provides an effective strategy to tailor pectin functionality for specific applications. Citrus pectins show strong potential for use in food systems such as emulsions, beverages, sauces, dressings, and low-fat formulations, where their hydration, stabilizing, and emulsifying properties are particularly advantageous [40].

3.9. Antioxidant Activity of Citrus Pectin

Citrus pectins exhibited relatively low antioxidant activity, with clear interspecific variation influenced by structural and compositional differences. The IC₅₀ values ranged from 438 to 460 µg/mL (Table 5), indicating weak radical-scavenging capacity compared with polyphenolic antioxidants, yet consistent with typical polysaccharide-based systems. Among the samples, Citrus limon and Citrus paradisi showed comparatively higher antioxidant activity (IC₅₀ = 438 and 440 µg/mL; TE = 340 and 334 mg/g, respectively), as detailed in Table 5. This enhanced performance is associated with their lower DM, which increases

the availability of free carboxyl and hydroxyl groups, facilitating hydrogen donation and improving electron-transfer efficiency. In contrast, *Citrus maxima* exhibited the lowest activity ($IC_{50} = 457 \mu\text{g/mL}$; $TE = 217 \text{ mg/g}$), likely due to its higher esterification level, which reduces reactive functional groups and limits metal-chelating and electrostatic interactions. Intermediate antioxidant behavior observed in *C. reticulata* and *C. sinensis* reflects a balance between esterified domains, neutral sugar content, and co-extracted compounds.

Table 5. Antioxidant capacity and anti-diabetic activity of citrus pectins.

Samples	Antioxidant (IC_{50} ($\mu\text{g/mL}$))	Antioxidant TE (mg/g)	Amylase IC_{50} ($\mu\text{g/mL}$)	Amylase AE (mg/g)
Acarbose	-	-	145 ± 5.0^c	975 ± 11^a
<i>Citrus aurantium</i>	460 ± 9.0^a	310 ± 5.3^e	337 ± 7.4^{ab}	496 ± 7.0^{bc}
<i>Citrus limetta</i>	445 ± 7.0^{ab}	330 ± 8.0^{bc}	338 ± 5.5^{ab}	490 ± 7.3^c
<i>Citrus limon</i>	438 ± 7.5^b	340 ± 5.5^b	330 ± 6.5^b	510 ± 7.6^b
<i>Citrus maxima</i>	457 ± 8.5^a	217 ± 5.0^{de}	335 ± 8.0^{ab}	504 ± 7.3^b
<i>Citrus paradisi</i>	440 ± 7.0^b	334 ± 6.6^b	345 ± 7.3^a	477 ± 5.5^c
<i>Citrus reticulata</i>	448 ± 6.7^{ab}	325 ± 7.3^{bc}	340 ± 7.5^{ab}	485 ± 5.7^c
<i>Citrus sinensis</i>	450 ± 8.0^{ab}	322 ± 6.0^{cd}	343 ± 7.0^{ab}	480 ± 6.5^c
Trolox	135 ± 4.0^c	985 ± 12^a	-	-

Antioxidant capacity is expressed as the half-maximal inhibitory concentration (IC_{50}) and Trolox equivalents (TE). Anti-diabetic activity against α -amylase is reported as IC_{50} and acarbose equivalents (AE). All values are presented as means $\pm$ standard deviation (SD) of three replicates ($n = 3$). Different superscript letters within the same column indicate significant differences according to Tukey's test ($p < 0.05$).

Despite their relatively low radical-scavenging potency, these findings align with previous reports indicating that non-starch polysaccharides generally exhibit modest antioxidant activity, primarily through hydrogen atom transfer and metal chelation rather than strong electron-donating capacity [54]. The antioxidant behavior of pectin is largely governed by its homogalacturonan backbone and rhamnogalacturonan regions, where accessible hydroxyl and carboxyl groups participate in radical neutralization. Low-molecular-weight and low-DM pectins typically demonstrate enhanced activity due to increased accessibility of reactive sites and greater conformational flexibility [55]. In addition, citrus pectin often retains bound or co-extracted polyphenolic compounds, such as flavonoids, which can contribute synergistically to antioxidant activity. These compounds provide aromatic systems capable of stabilizing radical intermediates and enhancing overall redox performance [56]. Pectin also exhibits metal-chelating properties, particularly toward Fe^{2+} and Cu^{2+} ions, thereby reducing the formation of reactive oxygen species via Fenton-type reactions [57].

Structural proteins co-extracted with pectin further influence antioxidant behavior. These proteins contain redox-active amino acids (e.g., tyrosine, tryptophan, cysteine, and methionine) that can participate in hydrogen atom transfer and electron-transfer reactions. Their interaction with the pectin matrix may enhance the exposure and mobility of functional groups, facilitating radical scavenging and metal binding. In low-DM pectins, these protein–polysaccharide interactions are more pronounced, contributing to improved antioxidant efficiency. Beyond direct antioxidant mechanisms, pectin may exert indirect effects in biological systems. Fermentation of pectin in the gastrointestinal tract produces short-chain fatty acids, which can modulate oxidative stress by enhancing endogenous antioxidant defense systems, including enzymes such as superoxide dismutase, catalase, and glutathione peroxidase [58]. Overall, the antioxidant activity of citrus pectin is structurally governed and arises from the combined effects of the degree of methylation, molecular architecture, and associated bioactive compounds. Low-DM pectins enhance hydrogen-donating and

metal-chelating capacity, while phenolics and protein residues provide additional redox-active sites. Neutral sugar side chains influence molecular flexibility and accessibility of reactive groups, further modulating antioxidant behavior. Although citrus pectin is a relatively weak direct antioxidant, its multifunctional properties and synergistic interactions make it a valuable component in functional foods and nutraceutical applications [59].

To place the obtained antioxidant activities in the context of the current State of the Art, the results were compared with previously reported citrus-derived pectins. The IC_{50} values obtained in the present study (438–460 $\mu\text{g/mL}$) were considerably lower than those reported for lemon peel pectins, which exhibited IC_{50} values ranging from 1019 to 1383 $\mu\text{g/mL}$ in DPPH radical-scavenging assays [60]. Similarly, citrus pectins isolated from citrus canning processing water showed IC_{50} values between 2.68 and 18.89 mg/mL , corresponding to substantially weaker antioxidant activity than that observed in the present work [61]. Nevertheless, the antioxidant capacity of the investigated pectins remains lower than that of conventional low-molecular-weight antioxidant standards such as ascorbic acid, which typically exhibits IC_{50} values in the low $\mu\text{g/mL}$ range [60]. These findings indicate that the citrus pectins extracted in this study possess antioxidant activity that is comparable to or stronger than that of many previously reported citrus pectin systems, highlighting their potential for application in functional food and nutraceutical formulations.

3.10. α -Amylase Inhibitory Activity of Citrus Pectin

Citrus pectins exhibited moderate α -amylase inhibitory activity, with IC_{50} values ranging from 330 to 345 $\mu\text{g/mL}$ (Table 5), which is lower than that of the pharmaceutical inhibitor acarbose ($IC_{50} \approx 145 \mu\text{g/mL}$). Nevertheless, the observed inhibition is consistent with previous reports demonstrating that dietary polysaccharides can modulate carbohydrate digestion and reduce postprandial glucose release. The inhibitory activity of citrus pectin is influenced by several structural parameters, including degree of methylation (DM), molecular weight, charge density, branching pattern, and the presence of associated phenolic compounds.

Although the present study did not include enzyme kinetic analyses necessary to determine the exact mode of inhibition, previous investigations suggest that pectins generally act through non-competitive or mixed-type inhibition mechanisms rather than classical competitive inhibition. In these mechanisms, pectin molecules interact with regions outside the catalytic site through hydrogen bonding, electrostatic interactions, and weak hydrophobic associations, inducing conformational changes that reduce catalytic efficiency [58,62].

In addition, pectin may decrease enzyme activity indirectly by increasing solution viscosity, limiting enzyme–substrate diffusion, and forming physical barriers around starch granules, thereby restricting substrate accessibility [63]. The inhibitory effect may also be enhanced by the ability of pectin to chelate divalent ions such as Ca^{2+} , which contribute to the structural stability of α -amylase [64]. Consequently, the observed inhibition likely results from a combination of direct enzyme–polymer interactions and indirect physicochemical effects rather than a single binding mechanism. Although less potent than pharmaceutical inhibitors, citrus pectins may contribute to glycemic control through delayed starch digestion and reduced glucose absorption. Their natural origin, dietary fiber functionality, and low risk of adverse effects make them attractive candidates for incorporation into functional foods designed for low-glycemic-index applications. Future studies employing enzyme kinetic analyses are required to definitively establish the inhibition mechanism and determine whether the observed activity follows non-competitive, mixed-type, or other inhibitory models [65].

4. Conclusions

This study provides a comprehensive comparison of pectins extracted from seven citrus species and demonstrates that, despite their similar biochemical composition, they exhibit distinct physicochemical, structural, thermal, techno-functional, and bioactive properties. The extracted pectins were predominantly amorphous, thermally stable over a broad temperature range, and exhibited negative zeta potentials, nanoscale particle sizes, and shear-thinning behavior, indicating favorable colloidal characteristics. Significant differences were observed in techno-functional properties, including swelling capacity, hygroscopicity, water- and oil-holding capacities, emulsifying behavior, and solubility, highlighting the influence of citrus species on pectin functionality. The pectins also showed moderate antioxidant and α -amylase inhibitory activities, suggesting potential applications in functional food formulations aimed at supporting glycemic control and oxidative stress management. Overall, the results demonstrate that citrus peel is a valuable and sustainable source of multifunctional pectin and that the selection of citrus species can be used to tailor pectin properties for specific food, pharmaceutical, and nutraceutical applications. Future studies should focus on structure–function relationships and targeted modifications to further enhance pectin performance in advanced applications.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/polysaccharides7030082/s1>. Figure S1: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus aurantium pectin; Figure S2: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus limetta pectin; Figure S3: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus limon pectin; Figure S4: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus maxima pectin; Figure S5: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus paradisi pectin; Figure S6: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus reticulata pectin; Figure S7: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus sinensis pectin; Figure S8: Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) of Citrus sinensis pectin; Figure S9: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus aurantium pectin; Figure S10: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus limetta pectin; Figure S11: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus limon pectin; Figure S12: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus maxima pectin; Figure S13: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus paradisi pectin; Figure S14: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus reticulata pectin; Figure S15: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus sinensis pectin; Figure S16: Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of Citrus sigma pectin.

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