

Comparative Acid Hydrolysis and Physicochemical Characterization of Agro-Food Processing Residues for Glucose-Rich Extract Production

Tuna Karataş

Sabancı University: Sabancı Universitesi

Deniz Saltık

Sabancı University: Sabancı Universitesi

İdil Ceylin Toğan

Sabancı University: Sabancı Universitesi

Berna Oruc

Kordsa Global

Duygu Ekinci

Kordsa Global

Gokce Ugur

Kordsa Global

Sahar Porrang

Sabancı University: Sabancı Universitesi

Nur Mustafaoglu

nur.mustafaoglu@sabanciuniv.edu

Sabancı University: Sabancı Universitesi <https://orcid.org/0000-0001-8219-9399>

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Abstract

Agro-food processing residues represent an abundant yet underutilized source of lignocellulosic carbohydrates for biorefinery applications. In this study, wheat husk, sweet lime peel, and carrot pomace were comparatively evaluated as potential feedstocks for glucose-rich extract production via dilute acid hydrolysis. The structural and compositional changes induced by hydrolysis were systematically investigated using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), inductively coupled plasma mass spectrometry (ICP-MS), and proton nuclear magnetic resonance (^1H NMR). All three residues showed evidence of lignocellulosic disruption and saccharide release after hydrolysis, with clear feedstock-dependent differences in hydrolysis behavior and extract composition. Among the tested residues, carrot pomace exhibited the most favorable overall performance, showing stronger glucose-related spectral features, reduced structural recalcitrance after treatment, and a mineral composition supportive of downstream bioprocessing potential. Sweet lime peel also demonstrated effective hydrolysis and selective glucose-associated signals, whereas wheat husk showed comparatively lower hydrolysis efficiency, consistent with its more rigid lignocellulosic structure. These findings provide a comparative physicochemical basis for selecting agro-food residues as carbohydrate-rich feedstocks and highlight carrot pomace as a promising candidate for glucose-oriented biomass valorization and future bioprocessing applications.

1. Introduction

Agro-food processing generates large quantities of lignocellulosic residues that are often underutilized despite their considerable biochemical potential. Globally, approximately 1.3 billion tons of food and agricultural waste are generated annually, contributing to environmental challenges such as greenhouse gas emissions and inefficient resource utilization [1, 2]. At the same time, these residues contain valuable carbohydrates, lignin, and other bio-based compounds that can serve as feedstocks for sustainable biochemical and bioindustrial applications [3, 4].

Conventional management strategies for agro-food residues, including landfilling and incineration, not only impose environmental burdens but also fail to exploit the intrinsic chemical value of these materials. Consequently, increasing attention has been directed toward the valorization of agricultural residues through biochemical conversion and biomass processing technologies. Methods such as enzymatic hydrolysis, steam explosion, and chemical pretreatment have demonstrated potential for recovering fermentable sugars and other value-added compounds from lignocellulosic biomass. However, many of these approaches remain limited by operational costs, scalability challenges, and complex processing requirements [5].

Among the target products derived from biomass processing, glucose is one of the most important platform molecules due to its wide-ranging industrial applications, including biofuel production [6], pharmaceuticals [7], and food processing [8]. Agro-food residues such as wheat husk (WH), sweet lime peel (SL), and carrot pomace (CP) represent promising carbohydrate sources because of their high cellulose and hemicellulose content. While wheat-derived residues have been extensively studied for sugar recovery [9–11], comparatively fewer studies have explored the hydrolysis potential of SL peel [12] and CP [13], particularly using dilute acid hydrolysis approaches [14].

In this study, we comparatively investigate WH, SL peel, and CP as potential feedstocks for glucose-rich extract production via dilute acid hydrolysis. Structural and compositional changes before and after hydrolysis were systematically examined using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA), inductively coupled plasma mass spectrometry (ICP-MS), and proton nuclear magnetic resonance (^1H NMR). Through this multi-technique characterization, the study aims to provide a physicochemical basis for assessing agro-food residues as carbohydrate-rich biomass resources for future bioprocessing and biomass valorization strategies.

2. Materials and Methods

2.1. Materials:

The chemicals and reagents used in this research were sulfuric acid (H_2SO_4 , Merck Chemicals) prepared as a 6% (w/w) solution for acid hydrolysis of biological waste samples. Distilled water (dH_2O) was used as washing, dilution and solvent throughout the experiment. Hydrogen peroxide (H_2O_2 , 30% v/v, Merck) was used to digest biological waste samples for Inductively Coupled

Plasma Mass Spectrometry (ICP-MS) analysis to ensure complete organic degradation. Nitric acid (HNO_3 , 65% v/v, Merck) was used in a closed-vessel microwave system to digest samples for elemental analysis. Ultrapure water (18.2 M Ω) was applied for all dilutions to minimise contamination and ensure accurate analytical measurements. These chemicals were crucial for performing hydrolysis, preparing samples and performing detailed characterizations using techniques such as Fourier Transform Infrared Spectroscopy (FTIR), XRD, Thermogravimetric Analysis (TGA) and ICP-MS.

2.2 Sample preparation

WHs are obtained from the Plant Nutrition and Physiology Laboratory of Sabanci University. WHs are separated from the stalk and additional waste. The separated WHs are washed with distilled water to remove dust and weighed by a scale (Radwag). SLs are obtained from local groceries in Istanbul. The peel is separated from the fruit with sterile knives. The separated lime peels are washed with dH_2O and weighed. The WH and SL peels are divided into plastic petri dishes and left to dry in a laboratory oven (BINDER) at 60°C for 24 hours. During drying, the WH and lime peels are weighed at different times until a constant weight value is obtained. The dried WHs and sweet lime peels are grinded with a coffee grinder (Bosch) for 70 and 100 seconds, respectively and stored at 4°C.

Carrots are obtained from local groceries in Istanbul. The carrots are washed with dH_2O and blended. The carrot juice is separated from the carrot pomace by sieving with cloth. 50 mL of carrot juice is stored in falcon flasks for pH analysis. The remaining carrot pomace are divided into three plastic petri dishes and weighed. The CP is dried in a laboratory oven at 60°C. Drying carrot pomaces are weighed at different times. The dried carrot pomace is grinded with a coffee grinder for 60 seconds and stored at 4°C. For the pH value of carrot juice is measured by a pH meter (Inesa REX).

2.3 Moisture analysis of biowaste samples

During drying, multiple weight measurements (at 0, 16, 19, 21, and 48 hours) are performed to ensure complete drying of each sample. The samples are periodically weighed until a constant weight value is obtained, indicating the removal of moisture. The moisture percentage (%) was calculated by subtracting the final (dry) weight from the initial weight, dividing the difference by the initial weight, and multiplying by 100.

2.4 Acid hydrolysis and post-treatment of biowaste samples

For acid hydrolysis of samples, a dilute H_2SO_4 (Merck) solution is prepared by mixing 6.522 mL of concentrated H_2SO_4 with 188 mL dH_2O . A silicone oil bath (Munro) is used to ensure high thermal stability and homogenous heating, thereby improving reaction uniformity. During the hydrolysis process, 200 mL of 6% (w/w) H_2SO_4 is transferred into a double necked flask. The flask is placed on a magnetic heater-stirrer (Witeg) set to 400 rpm, and the temperature is increased to 100°C using a heater set at 230°C. Once the acid solution reaches 100°C, as verified with a thermometer, 2 g of the biowaste powder is added. The mixture is maintained at 100°C for 60 minutes under continuous stirring. The same procedure is applied for acid hydrolysis of WH, SL peel, and CP.

After the reaction mixture cools to 50°C, it is centrifuged at 4700 rpm and 20°C for 20 minutes. Following the initial centrifugation, the pellet obtained from carrot pomace hydrolysis is washed with 20 mL dH_2O and centrifuged again under the same conditions. For the SL peel samples, centrifugation is extended to 40 minutes to ensure complete separation. After centrifugation, the supernatants are carefully removed, and the remaining pellets are dried in an oven (Binder) at 60°C for 24 hours and the supernatants and their pellets are separated and stored at 4°C for further analysis.

2.5 Analytical characterization of biowaste samples before and after acid hydrolysis

To evaluate the glucose-associated spectral signals achieved through acid hydrolysis of WH, SL peel, and CP biowastes, multiple analytical techniques are employed. These include FTIR, XRD, TGA, ICP-MS, and NMR spectroscopy, including both Carbon-13 (^{13}C NMR) and Proton (^1H NMR) analyses. Before and after acid hydrolysis, the biowaste samples are analysed using FTIR spectroscopy (Thermo Scientific™ NICOLET iS™ 10). XRD analysis was conducted using a D2 PHASER benchtop X-ray diffractometer (Bruker) operated with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) at 30 kV and 10 mA. The diffraction patterns were recorded over a 2θ range of 10°–90° under Ψ -tilting conditions, with a step size of 0.02° and a scan speed of 1°/min to identify the crystalline phases and structural changes in the samples. TGA is performed using a STA 449 C Jupiter® thermo-microbalance

from (Netzsch-Gerätebau GmbH) under an air atmosphere. Samples are analysed between 25°C and 800°C temperature with a heating rate of 10°C/min. For ICP-MS analysis, the biowaste samples are digested in a closed-vessel microwave system (MarsExpress CEM Corp) with 2 mL of 30% (v/v) H₂O₂ (Merck) and 5 mL of 65% (v/v) HNO₃ (Merck). All dilutions are prepared using ultra-pure water (18.2 MΩ). Elemental analysis is performed using Agilent Technologies 7700 Series ICP-MS system.

3. Results and discussion

3.1 Moisture analysis of raw samples

Determination of moisture content is essential for optimizing drying conditions and minimizing variability during biomass processing. Initially, the masses of SL peel and CP were 62.30 g and 65.43 g, respectively, which decreased to 13.67 g and 11.23 g after oven drying at 60°C for 48 hours. In contrast, WH exhibited substantially lower moisture content. A decrease in the rate of moisture loss was observed after approximately 16 hours of drying for both SL peel and CP, as illustrated in the normalized weight-loss profiles (Fig. 1A). Based on these observations, drying durations between 16 and 48 hours were considered adequate for achieving stable moisture removal in future experiments. The measured moisture contents of CP, SL peel, and WH were 82.83%, 78.05%, and 4.32%, respectively (Fig. 1B).

3.2 FTIR of raw samples

The FTIR spectrum of raw WH is presented in Fig. 2, and corresponding band assignments compared with previous studies are summarized in Supplementary Table 1. The band at 3319.76 cm⁻¹ is similarly observed by Wang et al. [15] at 3348 cm⁻¹ and Buazar et al. [16] at 3320 cm⁻¹, and assigned to O-H stretching vibrations. Javier-Astete et al. [17] further related this vibration to the existence presence of cellulose. The 2918.80 cm⁻¹ peak corresponds to C-H stretching in cellulose and hemicellulose, consistent with peaks reported by Wang et al. [15] at 2923 cm⁻¹ and [16] at 2915 cm⁻¹. The 1728.27 cm⁻¹ band, observed at 1738 cm⁻¹ by [16], is attributed to C = O stretching of hemicellulose. The 1631.15 cm⁻¹ band, seen by Wang et al. [15] at 1650 cm⁻¹, is assigned to O-H stretching and hydrogen bonding. The 1030.96 cm⁻¹ band, corresponding to C-O-C stretching typical of glucan in cellulose, aligns with those observed at 1041 cm⁻¹ [15] and 1012 cm⁻¹ by Buazar et al. [16]. The 790.98 cm⁻¹ band, similar to the 785 cm⁻¹ band reported by Buazar et al. [16], represents the stretching vibration of the silanol (Si-OH) groups present WH. The 1370.75 cm⁻¹ peak corresponds to C-H bonding in cellulose and hemicellulose, consistent with the findings of Javier-Astete et al. [17].

The FTIR spectrum of the raw SL peel is also shown in Fig. 2, with band assignments summarized in Supplementary Table 2. The band at 3303.29 cm⁻¹ aligns with those reported by Malakar et al. [12] and John et al. [18], corresponding to O-H stretching vibrations of cellulose. The 2919.86 cm⁻¹ peak, observed by Malakar et al. [12] at 2920.50 cm⁻¹ and John et al. [18] at 2901 cm⁻¹, is attributed to asymmetric C-H stretching of methyl and methylene groups of cellulose. The 1743.69 cm⁻¹ band, observed at 1733.97 cm⁻¹ [12] and at 1745 cm⁻¹ [18], is assigned to C = O stretching vibrations. Malakar et al., [12] associate this vibration with hemicellulose, whereas John et al. [18] attribute it to lignin. The 1608.59 cm⁻¹ band, comparable to 1603.72 cm⁻¹ [12] and 1606 cm⁻¹ [18], is related to C = O stretching of lignin. The 1435.53 cm⁻¹ and 1375.49 cm⁻¹ peaks are consistent with the 1434.98 cm⁻¹ and 1369.20 cm⁻¹ bands reported by Malakar et al. [12], indicating the presence of cellulose and hemicellulose, respectively. The 1013.90 cm⁻¹ band, along with the 1016 cm⁻¹ peak reported by John et al. [18], is assigned to the C-O, C-C, C-OH stretching vibrations characteristics of cellulose, hemicellulose, and lignin. Three unique peaks at 1233.98 cm⁻¹, 913.90 cm⁻¹ and 886.10 cm⁻¹ peaks are observed only in this study. The 1234 cm⁻¹ band is consistent with C-O-C / C-O stretching and ionized carboxyl/ester vibrations commonly observed in pectins and esterified polysaccharides (e.g., citrus pectins) [19]. According to Javier-Astete et al., the presence of 913.90 cm⁻¹ and 886.10 cm⁻¹ peaks may correspond to C1-H deformation in cellulose, due to contamination by cellulase-producing microorganisms [17].

The FTIR spectrum of the raw CP is shown in Fig. 2, with comparative data presented in Supplementary Table 3. The 3286.80 cm⁻¹ band, observed at 3330 cm⁻¹ by Mousavi et al. [20] and 3382.98 cm⁻¹ by Jafari et al. [21], is attributed to O-H stretching vibrations of cellulose. The 2933.07 cm⁻¹ peak, reported at 2920 cm⁻¹ by [20] and 2932.48 cm⁻¹ by [21], corresponds to C-H stretching of

methyl groups in cellulose, hemicellulose, and lignin [20] or solely in cellulose [21]. The 1729.66 cm^{-1} , observed at 1733 cm^{-1} [20] and 1742.97 cm^{-1} [21], signifies C = O stretching of carbonyl groups in lignin, pectin, hemicellulose. The 1604.56 cm^{-1} band, corresponding to 1607 cm^{-1} [20] and 1656.70 cm^{-1} [21], is attributed to C = O stretching vibration of free carboxyl groups in hemicellulose. The 1323.48 cm^{-1} band, similar to 1367 cm^{-1} [20], is associated with cellulose, while the 1234 cm^{-1} peak is assigned to the C-O stretching in lignin. The 1011.78 cm^{-1} peak corresponds to the amorphous region of cellulose, comparable to the 934.38 cm^{-1} band reported by [21]. Two additional peaks, at 2884.86 cm^{-1} and 2285.19 cm^{-1} , are observed exclusively in this study.

3.3 FTIR analysis of hydrolysed biowaste samples

The FTIR spectrum of the hydrolysed wheat husk (HWH) is presented in Fig. 2A, and comparative analysis with previous studies is summarized in Supplementary Table 4. Following acid hydrolysis, the FTIR results reveal biochemical alterations. For WH, the C-H stretching band shifts to 2916.17 cm^{-1} , indicating changes in cellulose and hemicellulose conformations [15]. The appearance of a new band at 1691.68 cm^{-1} suggests hemicellulose breakdown, while additional peaks at 1101.57 cm^{-1} and 1017.08 cm^{-1} correspond to glucose formation, confirming carbohydrate hydrolysis [22]. The peaks associated with lignin at 1631.15 cm^{-1} and 1462.36 cm^{-1} post-hydrolysis indicate modifications in lignin structures, which are critical for improving cellulose accessibility during bioconversion. Moreover, the appearance of a new band at 870.29 cm^{-1} indicates monosaccharide formation, essential for downstream biofuel fermentation processes [17].

The FTIR spectrum of the hydrolysed sweet lime peel (HSL) is also shown in Fig. 2A. After hydrolysis, notable changes are observed in the O-H stretching peak of 3357.08 cm^{-1} and the C = O stretching peak at 1707.07 cm^{-1} , indicating structural modifications in lignin and hemicellulose [12]. When compared with the FTIR spectrum of glucose reported by Adina et al. [23], similar absorption peaks are observed, suggesting successful glucose formation in acid HSL. The 1537.08 cm^{-1} band correspond to the C = C stretching of lignin, while the reduced intensities of hemicellulose-associated peaks indicate partial hydrolysis [23]. New absorption bands are detected at 1143.71 cm^{-1} , 1013.96 cm^{-1} , and 868.07 cm^{-1} . Comparable peaks reported by Adina et al. at 1149 cm^{-1} , 1107 cm^{-1} , and 1033 cm^{-1} are associated with glucose derived from cellulose, hemicellulose and lignin degradation [23] (Supplementary Table 5).

The FTIR spectrum of hydrolysed carrot pomace (HCP) is shown in Fig. 2A. Peaks observed at 1144.13 cm^{-1} , 1098.11 cm^{-1} , 1012.94 cm^{-1} , and 871.96 cm^{-1} correspond to glucose formation. These results closely align with those of Adina et al. [23], who reported glucose-associated bands at 1149 cm^{-1} , 1107 cm^{-1} , 1078 cm^{-1} , 1033 cm^{-1} , and 991 cm^{-1} (Supplementary Table 6). Additionally, the present findings are consistent with [24], who identified peaks at 1134 cm^{-1} , 1109 cm^{-1} , 1045 cm^{-1} , 987 cm^{-1} , and 920 cm^{-1} , corresponding to monosaccharide variations of glucose and fructose (Supplementary Table 6). These results confirm that glucose is successfully extracted from CP through acid hydrolysis treatment.

3.4 FTIR analysis of supernatants from acid hydrolysed sample

The FTIR spectra of the supernatants obtained from acid-hydrolysed samples reveal clear biochemical alterations, indicating the presence of residual polysaccharides and simple sugars, detailed band summary provided in Supplementary Table 7.

The FTIR spectrum of the hydrolysed wheat husk supernatant (WHE) is shown in Fig. 2A. The broad peak at 3355.74 cm^{-1} , attributed to O-H stretching, reflects the presence of hydroxyl-rich compounds such as cellulose and other alcohol-containing biomolecules that remain after hydrolysis. The band at 1635.18 cm^{-1} corresponds to C = O stretching vibrations commonly associated with carbonyl groups in lignin and hemicellulose, suggesting partial degradation or modification of these polysaccharides during acid treatment.

The FTIR spectrum of the hydrolysed sweet lime peel supernatant (SLE line) is also presented in Fig. 2A. The band at 3356.23 cm^{-1} , corresponding to the hydrogen-bonded O-H stretching, is consistent with that reported by Malakar et al. [12] at 3338.81 cm^{-1} and indicates the presence of cellulose and structural modification of cellulose due to hydrolysis (Supplementary Table 7). The 2929.48 cm^{-1} peak corresponds to C-H stretching of methyl and methylene groups in cellulose, also in agreement with Malakar et al. [12], confirming that this structural component remains partially intact in the supernatant. Additional bands observed at

2129.48 cm^{-1} and 1635.32 cm^{-1} correspond to C = O stretching vibrations typically found in lignin-carbohydrate complexes, again in line with previous findings [12]. These results indicate that hydrolysis of SL peel modifies the lignin network while partially preserving the cellulose and hemicellulose structures.

The FTIR spectrum of the hydrolysed carrot pomace supernatant (CPE) is displayed in Fig. 2A. New peaks appearing at 1634.32 cm^{-1} , 1192.03 cm^{-1} , 1050.14 cm^{-1} , and 587.11 cm^{-1} , which were absent in the raw CP, indicate the presence of glucose and other low-molecular-weight saccharides formed during hydrolysis. Koyama et al. [25] reported similar glucose associated peaks at 1155 cm^{-1} , 1090 cm^{-1} , and 1110 cm^{-1} . Likewise, Song et al. [26] observed corresponding peaks at 3379 cm^{-1} , 2360 cm^{-1} , 1653 cm^{-1} , and at 1066 cm^{-1} , also linked to glucose vibrations. Since these peaks are only observed in the hydrolysed CP and its supernatant, it can be concluded that glucose is successfully extracted from CP through acid hydrolysis.

3.5 XRD analysis of raw samples

XRD analysis of raw WHS, raw SL, and raw CP reveals both crystalline and amorphous characteristics of the biomass, supporting their potential suitability for bioconversion (Fig. 2B).

The diffractogram of raw WH exhibits a major peak around 2θ of 20° , which corresponds to the amorphous cellulose structure characteristic WH, as reported by Terzioglu et al. [27]. This suggests that cellulose is a predominant component of the raw WH. Minor peaks appearing near 2θ of 15° and 2θ of 34° (Fig. 2B) may indicate the presence of crystalline cellulose fractions, also consistent with the findings of Terzioglu et al. [27].

The diffractogram of the raw SL peel displays distinct peaks at approximately 2θ of 20° and 35° indicative of an amorphous cellulose structure (Fig. 2B). There are also several smaller peaks are also observed between 35° and 50° , suggesting the coexistence of crystalline domains within a predominantly amorphous cellulose matrix. These features align with compositional characteristic reported for citrus biomass by John et al. [18].

The diffractogram of the raw CP shows a major crystalline peak at 2θ of 15.7° (Fig. 2B), consistent with peaks reported at 16° by Rezvani et al. [28] and 15° by Wang et al. [29]. Another prominent crystalline peak appears at 2θ of 21.7° , which matches the peak at 21° documented by both Rezvani et al. [28] and Wang et al. [29]. These diffraction features are attributed to the cellulose I polymorph, typical plant-derived cellulose. Additionally, a minor peak is detected near 2θ of 12.1° , while another at 2θ of 34.6° corresponds to similar peaks reported by Rezvani et al. [28] and Trache et al. [30]. The former may be associated with pectin, while the latter can also be linked to cellulose I. The diffractogram of CP exhibits a broad amorphous region, indicating a semi-crystalline structure characteristic of heterogeneous lignocellulosic biomass.

3.6 XRD analysis of hydrolysed samples

The diffractogram of acid HWH exhibits two minor peaks around 2θ of 18° and 2θ of 30° (Fig. 2B). The major peak observed at 2θ of 20° in the raw WH becomes significantly broadened and diminished following hydrolysis. The appearance of these minor peaks and the broadening of previously sharp reflections indicate a notable reduction in crystallinity. This loss of crystallinity, a typical outcome of acid treatment, reflects the disruption of the ordered cellulose microfibrils and the breakdown of hydrogen bonding within the lattice [31]. Additionally, the minor peaks near 2θ of 15° and 2θ of 34° in raw sample become less distinct and show reduced intensity (Fig. 2B). These reflections, generally associated with crystalline regions of cellulose and hemicellulose, suggest structural degradation of these compounds during acid hydrolysis. The resulting increase in amorphous content enhances the biomass accessibility to enzymatic attack, demonstrating that acid hydrolysis effectively prepares WHs for subsequent bioconversion processes, such as enzymatic saccharification [31].

The diffractogram of the HSL peel displays major peaks at approximately 20° , 30° and 45° , but with noticeably reduced intensity compared to the raw SL peel (Fig. 2B). This attenuation suggests a decrease in crystallinity due to acid hydrolysis, which likely disrupts hydrogen bonding in cellulose and hemicellulose chains, leading to a more amorphous structure. The preservation of peak positions indicates that the fundamental crystalline framework remains, through in a less ordered form. The XRD patterns also reveals a broader amorphous region in the treated sample, supporting the transition from semi-crystalline to predominantly amorphous morphology as a result of the hydrolysis process.

The diffractogram of HCP is also shown in Fig. 2B. The major crystalline peak observed at 2θ of 17.3° corresponds to the band reported by Gupta et al. [32] at 2θ of 17° , which is associated with lignin. Another major peak appears at 2θ of 23° , comparable to the diffraction peak of powdered glucose at 2θ of 20° reported by Gu et al. [33], suggesting the formation of glucose after hydrolysis. A broad amorphous region is observed in the diffractogram, indicating that the treated CP display higher intensity than the raw sample, confirming the partial breakdown of ordered cellulose and lignin regions during hydrolysis.

The minor peaks at 2θ of 13° and 29.5° resemble the glucoglucose-associated peaks observed by Gu et al. (2017) at 2θ of 14° and 27° , while the peak at 2θ of 35° (Fig. 2B) aligns with those reported by Rezvani et al. [28] and Gong et al. (2017), corresponding to pectin and cellulose I, respectively. Collectively, these findings suggest that lignin and cellulose I structures identified in raw CP diffractogram are depolymerized into glucose following acid hydrolysis.

3.7 TGA of raw samples

TGA results of the raw WH, SL peel, and CP reveals distinct thermal degradation patterns that reflects fundamental differences in their lignocellulosic compositions (Fig. 3).

For WH, mass loss begins around 250°C , marking the onset of hemicellulose and cellulose decomposition, and continues up to approximately 400°C , where a prominent degradation phase is observed (Fig. 3A). This thermal behaviour indicates that WH possesses relatively high thermal stability, likely attributed its structured cellulose-lignin matrix, which enhances resistance to thermal degradation [34].

The TGA curve of raw SL peel shows an initial gradual mass loss starting near 50°C , corresponding primarily to moisture evaporation. A major degradation event occurs between 250°C and 350°C , associated with the thermal decomposition of cellulose and hemicellulose as well as the partial breakdown of the lignin network (Fig. 3B). The early onset of water loss compared to WH reflects the higher inherent moisture content of SL peel, a factor that may influence its pretreatment and processing requirements.

For raw CP, three main weight-loss stages are observed, corresponding to multi-step thermal decomposition (Fig. 3C). The first weight stage, between 100 – 190°C , corresponds to the release of moisture and light volatiles [35]. The second stage, occurring between 200 – 310°C , is attributed to the decomposition of polysaccharides such as cellulose, hemicellulose and lignin [36]. The third stage, between 360 – 800°C , involves oxidative degradation and the evolution of CO and CO₂ gases [35]. These results are consistent with the findings of Elkhailifa et al. [37], who reported similar weight-loss for carrot waste between 200 – 320°C and 320 – 500°C . However, the present results differ from those of Mousavi et al. [20], who observed decomposition peaks at 160 – 180°C , 180 – 280°C , and 300 – 600°C . Such discrepancies may arise from variations in sample loading, buoyancy effects, or fluctuations in the balance mechanism during analysis.

3.8 TGA of acid-hydrolysed samples

TGA of the acid-hydrolysed samples reveals significant changes in thermal degradation behavior compared to the raw biomass, indicating alterations in structural integrity and chemical composition induced by the acid treatment (Fig. 3)

For HWH, the onset of weight-loss occurs at approximately 200°C and continues up to 500°C , beginning at a markedly lower temperature than in the raw sample. The earlier onset of degradation suggests a reduction in thermal stability and loss of structural rigidity resulting from acid hydrolysis. The broader and more gradual degradation range further demonstrates the progressive breakdown of hemicellulose and partial depolymerization of cellulose, which enhances the reactivity of the biomass (Fig. 3A). Such extended degradation behavior reflects both chemical and physical modifications of the cellulosic structure caused by acid treatment, consistent with observations made for alkali-treated biomass by Irfan et al. [31]. The acid treatment's effectiveness is also reflected in the decrease in crystallinity and increase in amorphous content, leading to cellulose that is more susceptible to decomposition at lower temperatures [34]. This thermal behaviour confirms that acid hydrolysis enhances the reactivity and accessibility of the WH for downstream applications, such as enzymatic saccharification or biofuel production. However, in contrast to Irfan et al. [31], who distinct multi-stage degradation below 400°C , the present study exhibits a broader degradation range, likely due to differences in acid type, concentration, or reaction conditions.

For the HSL peel, the TGA curve shows a distinct thermal behavior compared to the raw sample. A noticeable mass loss begins near 100°C, attribute to moisture evaporation, which occurs at a significantly lower temperature than in the untreated sample, implying reduced structural cohesion and lower thermal stability (Fig. 3B). The main degradation phase to a higher temperature region, between 300°C to 400°C, corresponding to the thermal decomposition of carbohydrates and other organic constituents. This shift indicates the formation of simpler, less thermally stable degradation products as a result of the acid treatment. The faster mass loss during this phase suggests a more efficient decomposition process, likely due to the increased accessibility and depolymerization of biopolymers following hydrolysis. At temperatures above 600°C, the hydrolysed peel exhibits a noticeable lower residual mass, indicating more complete combustion and potentially a reduced mineral or ash content. These results align with the expected thermal behavior of acid-pretreated lignocellulosic materials, where the breakdown of structural polymers leads to decreased thermal resistance and altered degradation kinetics.

For HCP, two major weight-loss regions were observed. The first stage, between 100–250°C, corresponds to moisture evaporation and the release of light volatiles [35]. The second stage, extending from 320–800°C, is associated with the thermal decomposition of lignin, hemicellulose and cellulose into glucose [36]. Compared with the raw carrot pomace, the hydrolysed sample begins to decompose at a lower temperature (Fig. 3C), confirming that acid hydrolysis decreases the thermal stability of the material. Consequently, the raw CP demonstrates greater heat resistance and overall thermal stability than its hydrolysed counterpart.

3.9 ICP-MS analysis of acid-hydrolysed samples

The ICP-MS analysis of acid HWH, HSL, and HCP revealed distinct variations in their elemental composition (Table 1). Potassium (K) is the most abundant element across all samples. HCP exhibits a particularly high potassium concentration (1249 mg/kg), followed by HSL peel (1549 mg/kg) and HWH (296 mg/kg). This elevated K level suggests that CP may be especially suitable for biochemical applications where potassium-rich biomass enhances catalytic or fermentation processes.

Calcium (Ca) content is highest in SL peel (458 mg kg⁻¹), exceeding the concentrations in CP (184 mg kg⁻¹) and WH (132 mg kg⁻¹). This enrichment aligns with the known mineral profile of citrus residues, which typically contain substantial calcium in their cell-wall matrices. Magnesium (Mg) levels remain relatively consistent, with CP, SL peel, and WH containing 74, 54, and 109 mg kg⁻¹, respectively.

Table 1 ICP-MS analysis of acid-hydrolysed samples												
Sample	K mg/kg	Ca mg/kg	P mg/kg	Mg mg/kg	Na mg/kg	Al mg/kg	Fe mg/kg	Zn mg/kg	Mn mg/kg	Cu mg/kg	B mg/kg	Mo mg/kg
HCP	1249	184	140	74	151	22.20	9.70	1.20	0.38	0.50	3.49	0.31
HSL	1549	458	49	54	13	29.10	18.30	3.80	4.37	0.33	1.83	0.27
HWH	296	132	129	109	20	1.80	4.80	1.80	0.70	0.71	2.77	0.16

In terms of trace elements, HCP shows a balanced micronutrient profile, containing 9.70 mg kg⁻¹ iron (Fe) along with its high potassium content. HSL peel, however, displays the highest iron concentration among all samples (18.30 mg kg⁻¹), indicating potential for use in processes that benefit from Fe-rich biomass.

Overall, the ICP-MS results highlight the unique elemental signatures of each biowaste material. Among them, CP emerges as the most promising candidate due to its high potassium concentration, balanced micronutrient composition, and moderate levels of calcium and iron, which together suggest potential for various biochemical and catalytic applications. Conversely, the elevated calcium and iron contents in SL peel indicate its suitability for metal-dependent biochemical or adsorption processes (Table 1).

3.11 ¹H NMR of acid-hydrolysed samples

NMR analysis of acid-hydrolyzed biowaste supernatants

NMR spectroscopy is a powerful technique for elucidating the covalent structure of small organic molecules in solution. The ¹H NMR spectra of hydrolysed supernatants from wheat husk (WHE), sweet lime peel (SLE), and carrot pomace (CPE) provide detailed

insights into their molecular compositions (Fig. 4). All three spectra exhibit major peaks around 1.93 ppm and 3.54 ppm, indicating the presence of various sugar products. Additionally, a distinct peak near 3.19 ppm is observed in both the WH and CP supernatants, suggesting greater chemical diversity in these samples. The signal at approximately 1.93 ppm is attributed to acetic acid (CH_3COOH), a common product of lignocellulosic hydrolysis [38]. This peak also aligns with the findings of Cazor et al. [39], who report that this chemical shift may represent gamma-aminobutyric acid (GABA) in addition to acetic acid. The presence of acetic acid confirms partial deacetylation of hemicellulose, which is expected during acid hydrolysis and contributes to the formation of volatile organic acids that lower the pH of the hydrolysate. Both the WH and CP supernatants' spectra display additional peaks between 3.0-3.1 ppm, also linked to GABA [39].

The glucose resonance appears consistently between 3.2–3.3 ppm across all supernatants, confirming glucose formation [39]. Minor peaks observed around 3.9 ppm in the WHE and CPE spectra correspond to sucrose and fructose, with the signal at 3.82 ppm specifically correlating with sucrose [39]. The hydrolysed CP supernatant exhibits a distinct cluster of glucose-related peaks near 3.4 ppm [39]. The spectral region between 3.2 and 4.2 ppm closely resembles that of L-glucose, indicating that CP contains a higher glucose concentration compared to the other biowastes.

Furthermore, the 3.0 to 4.5 ppm region in all spectra, correspond to protons attached to C5, C2, C4, and C5 of anhydroxylose units found in arabinose and xylose [40]. Additional resonances identify several hydrolysis products: i) β -D-glucose, characterized by proton signals at δ 3.2, 3.4–3.59, 3.71–3.86, and 4.66 ppm [41]; ii) D-xylose, a known product of hydrolysis, with characteristic shifts at δ 3.6–3.68, 3.73–3.91, and 4.0 ppm [42]; iii) cellobiose, indicated by resonances at δ 3.2, 3.4–3.59, and 3.71–3.86 ppm [43]; and iv) L-arabinose, identified by peaks at δ 3.44–3.5, 3.59–3.67, 3.75–4.1, 4.48–4.5, and 5.2 ppm [44].

Among the three hydrolysed samples, CP shows higher peak intensities, confirming a more efficient release of glucose and other saccharides. The SL peel supernatant displays strong glucose-specific peaks with minimal interference from other sugars, indicating a glucose-associated spectral signals. In contrast, the WH supernatant exhibits weaker signals overall, consistent with lower hydrolysis efficiency due to its more rigid lignocellulosic matrix.

Overall, the ^1H NMR analysis confirms that acid hydrolysis successfully produces glucose and organic acids such as acetic acid from all three biowaste sources. Among them, CP achieves the most efficient sugar extraction, while SL peel yields a purer glucose extract. The detection of acetic acid in all spectra further validates the partial deacetylation of hemicellulose, a characteristic indicator of effective biomass hydrolysis.

4. Conclusion

This study comparatively evaluated wheat husk, sweet lime peel, and carrot pomace as agro-food processing residues for glucose-rich extract production through dilute acid hydrolysis. Combined FTIR, XRD, TGA, ICP-MS, and ^1H NMR analyses demonstrated that acid hydrolysis induced substantial structural and compositional changes in all three feedstocks, confirming lignocellulosic disruption and the release of soluble saccharide products.

Among the investigated residues, carrot pomace showed the most favorable overall response to hydrolysis, with stronger glucose-associated spectral features, reduced structural recalcitrance, and a balanced mineral profile. Sweet lime peel also exhibited efficient hydrolytic conversion and relatively selective glucose-related signals, while wheat husk showed lower hydrolysis efficiency, likely due to its more rigid lignocellulosic matrix. These comparative differences underline the importance of feedstock composition in determining hydrolysis behavior and carbohydrate accessibility.

Overall, this work provides a physicochemical framework for assessing agro-food residues as carbohydrate feedstocks and identifies carrot pomace as a promising raw material for glucose-oriented biomass valorization. The findings may support future studies focused on improving sugar recovery and integrating such residues into downstream bioprocessing and bioindustrial applications.

Declarations

Declaration of Competing Interest

Gokce Ugur reports a relationship with Kordsa Global that includes: employment. Berna Oruc and Duygu Ekinci previously employed by Kordsa Global. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data Availability

All data generated or analyzed during this study are included in this published article or are available from the corresponding author upon request.

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Figures

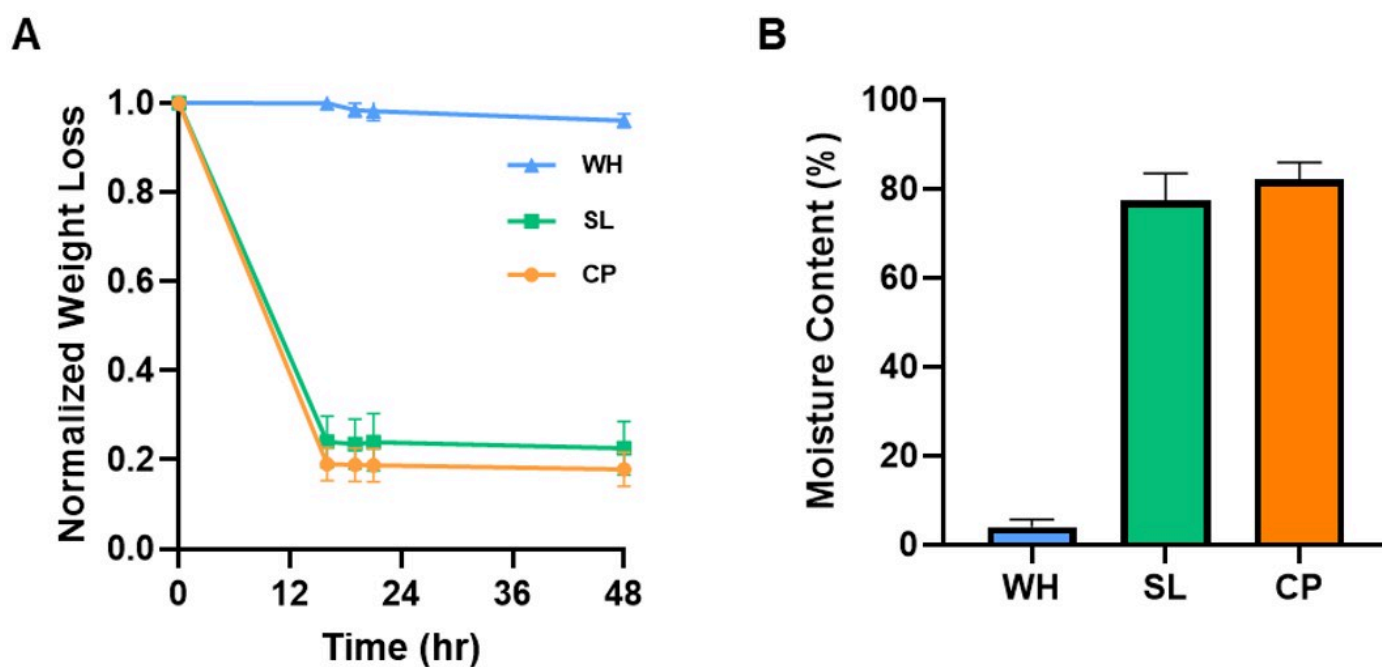


Figure 1

(A) Normalized weight loss of biowaste samples over 48 h at 60 °C. **(B)** Moisture content of the same samples. WH: Wheat Husk, SL: Sweet Lime Peel, CP: Carrot Pomace. All experiments were performed in triplicate. Error bars represent the standard deviation from the mean.

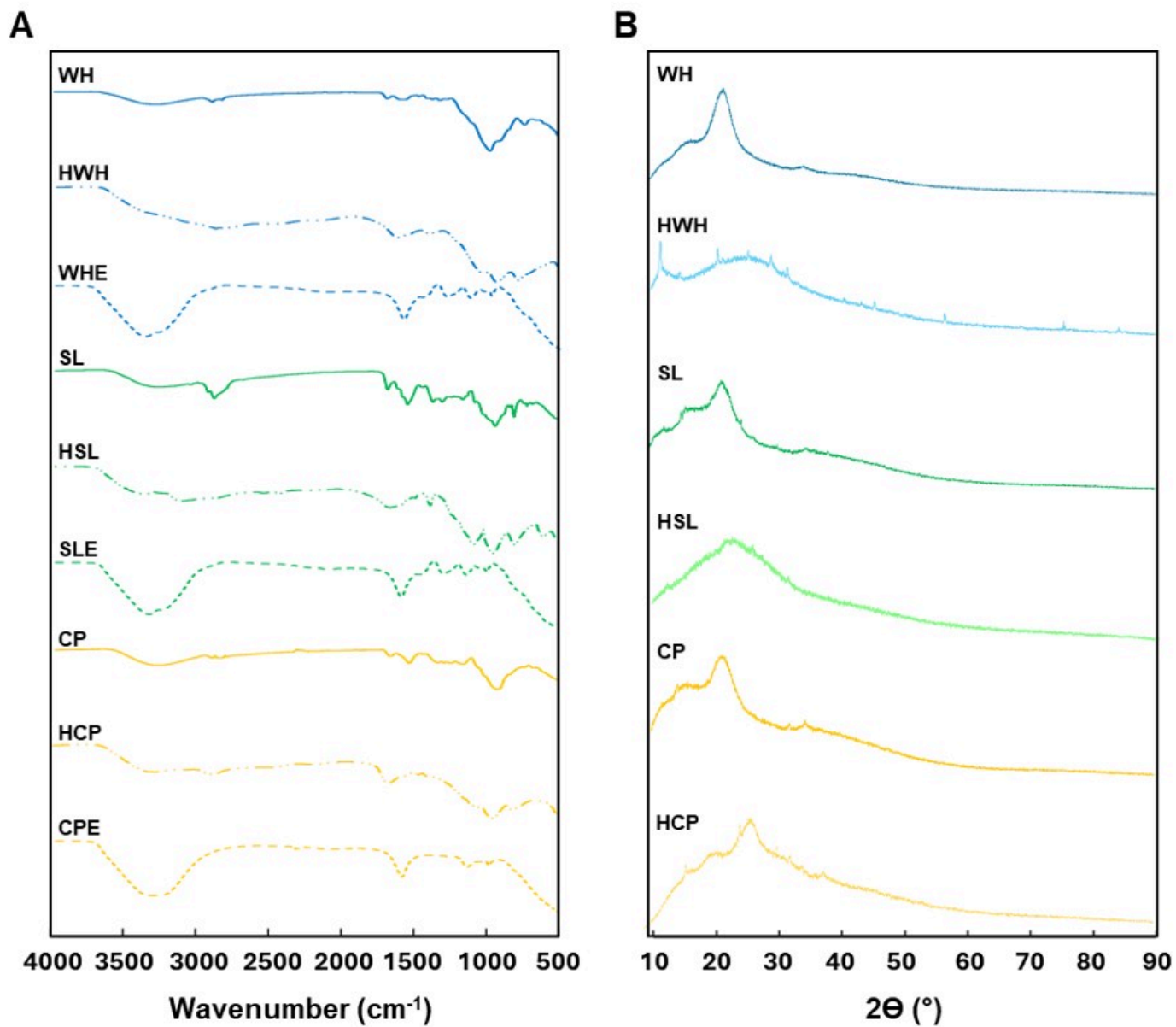


Figure 2

(A) FTIR and (B) XRD patterns of the same samples before and after hydrolysis. WH: Wheat Husk, HWH: Hydrolysed Wheat Husk, WHE: Wheat Husk Extract, SL: Sweet Lime Peel, HSL: Hydrolysed Sweet Lime, SLE: Sweet Lime Extract, CP: Carrot Pomace, HCP: Hydrolysed Carrot Pomace, and CPE: Carrot Pomace Extract.

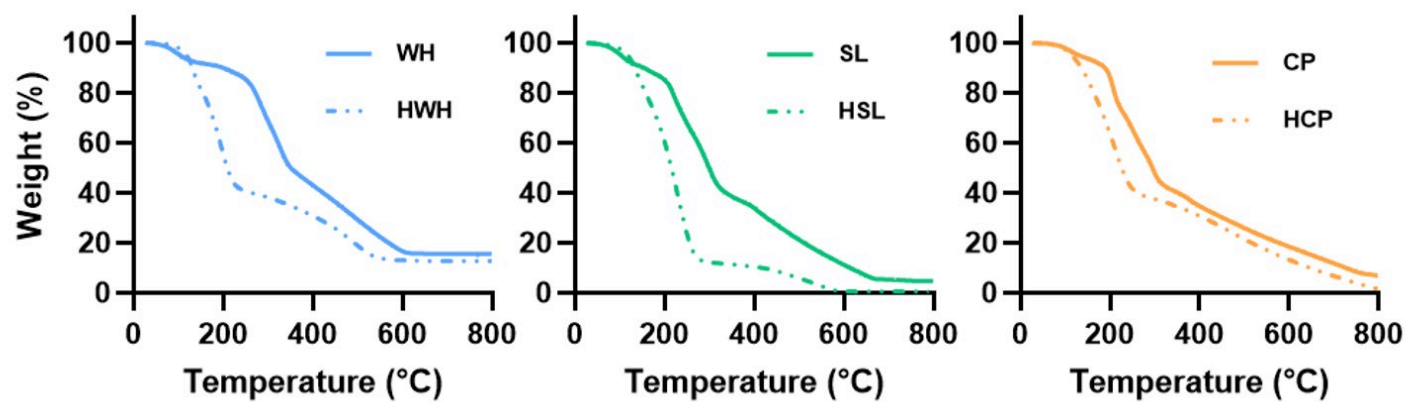


Figure 3

TGA of raw and acid-hydrolysed samples. WH: Wheat Husk, HWH: Hydrolysed Wheat Husk, SL: Sweet Lime Peel, HSL: Hydrolysed Sweet Lime, CP: Carrot Pomace. and HCP: Hydrolysed Carrot Pomace.

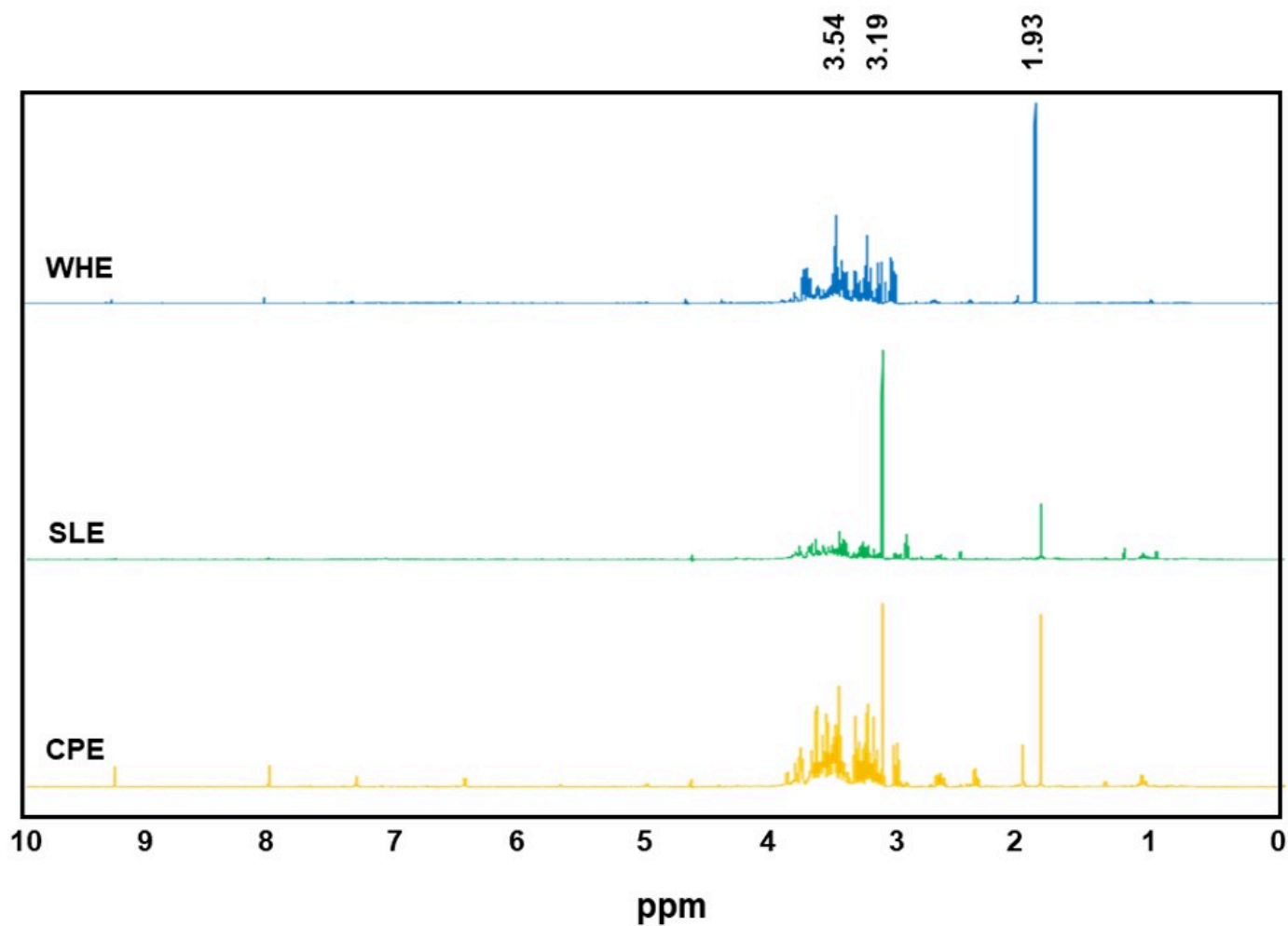


Figure 4

¹H NMR spectra of extracts obtained from wheat husk (WHE), sweet lime peel (SLE), and carrot pomace (CPE).

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