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Fourier Transform Infrared and Physicochemical Analyses of Roasted Coffee

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Supporting Information

ABSTRACT: In this study, Brazilian coffee beans processed to different stages of roast at 210, 220, 230, and 240 °C were analyzed for pH value, titratable acidity, moisture content, and color lightness. Fourier transform infrared (FTIR) spectroscopy, in conjunction with principal component analysis, was conducted to study the effects of process time and temperature on the IR-active components of the acetyl acetate extract of the roasted coffee. The results showed that high-temperature−short-time resulted in higher moisture content, higher pH value, and higher titratable acidity when the beans were roasted beyond the start-of-second-crack stage, as compare to low-temperature−long-time process (LTLT). The LTLT process also resulted in greater IR absorbance for aldehydes, ketones, aliphatic acids, aromatic acids, and caffeine carbonyl bands on the FTIR spectra. Clusters for principal component score plots were well separated, indicating that the changes IR-active components in the coffee extracts, due to the different roasting treatments, can be discriminated by the FTIR technique. On the basis of the loading plots of principal components, changes of IR-active compounds in the coffee extract at various stages of roasting were discussed.

KEYWORDS: *roasting profiles, coffee properties, FTIR, principal component analysis (PCA)*

INTRODUCTION

Green coffee beans provide neither the characteristic aroma nor the taste of a cup of coffee. To reveal their flavor, green coffee beans need to be roasted. Roasting is one of the most important steps in coffee processing that leads to the development of the desired aroma, taste, and color of the final brewed product. In general, the use of a roasting temperature of >200 °C is required to result in desirable chemical, physical, structural, and sensorial changes in the coffee beans.[1−3]

The time and temperature conditions applied during roasting have a major impact on the physical and chemical properties of roasted coffee beans. Geiger et al. reported that CO_2, a byproduct formed due to Strecker reactions and the degradation of organic compounds, increased greatly toward the end phase of a high-temperature−short-time process (260 °C, 170 s; HTST), whereas much lower amounts of CO_2 were formed when a low-temperature−long-time (228 °C, 720 s; LTLT) process was employed.[4] Schenker et al. found that a roasting process that involved a ramping temperature profile (from 150 to 240 °C in 270 s; 240 °C for 55 s) resulted in the formation of a greater quantity of aroma volatiles than a LTLT process (isothermal heating at 220 °C for 600 s).[3] Baggenstoss also reported that HTST roasting led to beans of lower density and higher volume, less roast loss, and lower moisture content as compared to the LTLT process.[2] Lyman et al. roasted green coffee beans under various process conditions to study the effect of roasting on brewed coffee.[5] Using a medium roast process (6.5 min to the onset of the first crack and 1.0 min to the onset of the second crack), Lyman et al. observed that coffee of balanced taste and aroma with citrus flavor was produced. However, using the so-called "sweated process" (4.5 min to the first crack and 6.5 min to the second crack), coffee beans of nonuniform bean color with "sour, grassy, and underdeveloped" properties resulted. In comparison, the "baked process" (11 min to the first crack and 18 min to the second crack) produced coffees that were "flat, woody with low brightness and acidity".[5] On the basis of these observations, one can conclude that the quality of roasted coffee does not solely depend on the physical parameters at the start and end points of roasting, but rather it is dependent on the time−temperature conditions applied during the roasting process.

Chemometrics employ statistical and mathematical techniques to convert complex spectral and chromatographic data into information with reduced dimensionality to facilitate interpretation.[6] Chemometric methodologies, such as principal component analysis (PCA), principal component regression, partial least-squares regression, and artificial neural network, have been successfully applied in process monitoring, detection of product adulteration, quality evaluation, screening of defective green coffee beans, and shelf-life study.[6−12] Although gas chromatography−mass spectrometry, gas chromatography, and sensory array instruments (electronic noses and tongues) have been used for studying the aroma compounds in roasted coffee,[13−15] analyses involving these techniques are time-consuming, and some of these instruments are complicated. Fourier transform infrared (FTIR)-attenuated total reflection (ATR) spectroscopy is a simple technique that is rapid and provides an overall infrared fingerprint of the specimen. Using an FTIR-ATR technique, Lyman et al. investigated the 1800−1680 cm^{-1} region of the IR spectrum of coffee brews. The carbonyl stretching region provided compositional information that can be used to correlate vinyl esters/lactones, esters,

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aldehydes, ketones, and acids.[5] In our previous study, FTIR-ATR spectroscopy was used as a tool for the rapid discrimination of the geographical origin of roasted coffee extracts and to study the changes in the FTIR spectra when coffees were roasted to different degrees.[16]

In the study, we applied a chemometric technique to analyze FTIR-ATR fingerprints of coffee beans at different stages of roast. The objectives of this study are to (1) develop the understanding of the effects of time−temperature conditions on the physical and chemical properties of coffee and (2) analyze coffees roasted to the same stage by different roasting profiles using the FTIR-ATR technique.

MATERIALS AND METHODS

Chemicals and Materials. Ethyl acetate was purchased from Fisher Scientific (Ottawa, Canada), ethanol from Greenfield Ethanol Inc. (Brampton, Canada), and sodium hydroxide from Sigma-Aldrich (Ontario, Canada). Wet-processed green coffee beans (Arabica) from Brazil were donated by Mother Parkers Tea & Coffee Inc. (Mississauga, Canada).

Green Beans and Roasting Conditions. Green coffee beans (45 g) were roasted in a fluidized bed hot air roaster (Fresh Roast SR 500, Fresh Beans Inc., Park City, UT, USA). Four isothermal roasting programs were used for roasting (Table 1). Coffee beans were collected at six roast stages (start of first crack, end of first crack, 48 s after first crack, start of second crack, end of second crack, and 48 s after second crack) and air-quenched to room temperature. The bean samples were stored in hermetic glass bottles in the dark at 15 °C before grinding.

Table 1. Time Taken To Achieve First and Second Cracks at Four Different Final Roast Temperatures

	time taken (min)			
final roasting temp (°C)	start of first crack	end of first crack	start of second crack	end of second crack
210	3.8	5.3	16.9	19.1
220	2.7	3.6	8.8	11.2
230	2.4	3.3	4.9	5.9
240	1.4	2.6	3.6	3.9

Degree of Roast As Determined by Color Measurements. Roasted coffee beans were ground using a coffee grinder (Bodum Antigua Electric Burr Grinder, Bodum, Inc., Copenhagen, Denmark), which was set at the medium level. The color of the ground coffee was measured in the L^*, a^*, b^* (Hunterlab) system using a Konica Minolta CM-3500d spectrophotometer (Konica Minolta Sensing, Inc., Osaka, Japan) in the reflectance mode. Before analysis, the instrument was calibrated on a white standard tile. Measurements were taken in triplicate.

Moisture Content of Ground Coffee. Gravimetrical determination of the moisture content of ground coffee was carried out using an oven dehydration method according to the Swiss Food Manual (SWFOH 1973). Samples of roasted beans were ground at the medium grind level with the Bodum Antigua Electric Burr Grinder and then dried at 103 °C for 5 h. Measurements were taken in triplicate.

pH Value. Two grams of medium-ground coffee was accurately weighed into a 200 mL glass bottle, and 100 mL of deionized water was added. The glass bottle was boiled for 10 min, and the mixture was filtered through Waterman qualitative filter paper. Then, 50 mL of the filtered extract was used for pH value determination with a pH-meter (Fisher Scientific, Accumet XL20, Ottawa, Canada). Measurements were taken in triplicate.

Titratable Acidity. Ten grams of medium-ground coffee was accurately weighed into a 200 mL glass bottle, and 75 mL of 80% ethanol was added to wet the sample. The glass bottle was shaken for 16 h under magnetic stirring, and the mixture was filtered through Waterman qualitative filter paper. After that, 50 mL of the filtered extract, filtered by Waterman qualitative filter paper, was titrated against 0.1 N NaOH solution to pH 8.2.[17] Measurements were taken in triplicate.

Solvent Extraction and ATR-FTIR Analysis of Ground Coffee. After medium grinding, coffee grounds were extracted with ethyl acetate following the extraction procedure: 0.2500 g of medium-ground coffee was accurately weighed into a glass vial, and 1 mL of deionized water was added to wet the sample. The glass vial was shaken for 1 min; 1 mL of ethyl acetate was added, and the mixture was shaken for an additional 5 min. The coffee extract was scanned using an FTIR spectrometer (IR Prestige-21; Shimadzu Corp., Tokyo, Japan) equipped with a deuterated triglycine sulfate detector and a KBr beam splitter. A MIRacle ATR accessory equipped with a diamond crystal (Pike Technologies, Madison, WI) was used for sampling. Before scanning, a drop of the extract (6 μL) was placed onto the ATR crystal, and the solvent was allowed to evaporate. The time required for the solvent to evaporate was determined by monitoring the spectrum until the solvent bands were no longer detectable. To collect the IR spectrum, samples were scanned from 600 to 4000 cm^{-1} at 4 cm^{-1} resolution, and 20 scans were averaged to give the final spectrum. All spectra were recorded at room temperature (23 ± 0.5 °C).[16] All extractions were performed in triplicate.

Chemometric Analysis. For chemometric analysis, raw FTIR spectra were exported as ASCII format, organized in Excel spreadsheets, and then analyzed using Pirouette v.4.0 software (Woodinville, WA, USA). A principal component analysis (PCA) was adopted to reduce the dimensionality of the FTIR data, as well as to facilitate the visualization of the data structure. Second derivative and mean center were applied to FTIR spectra to reduce baseline variation and enhance spectral features.

Scanning Electron Microscopy (SEM) Analysis. Coffee bean samples were cut perpendicular to the crevice to expose the internal bean structure. The bean samples were coated with a thin layer of gold in a sputter coater (model K550, Emitech, Ashford, Kent, U.K.). The pore structure of coffee beans was examined using a scanning electron microscope (SEM S-570, Hitachi High Technologies Corp., Tokyo, Japan) at an accelerating voltage of 10 kV. Micrographs were collected using image acquisition software (Quartz PCI, Version 7, Quartz Imaging Inc., Vancouver, BC).

RESULTS AND DISCUSSION

Evolution of Physical and Chemical Properties during Roasting. Roasting causes the buildup of pressure within the coffee bean due to the formation of steam and other gases. The increased internal pressure causes the bean to expand and crack, resulting in a phenomenon known as the "first crack". As heating continues to higher temperatures (>160 °C), the beans become darker and generate a large amount of CO_2, with concomitant degradation of carbohydrates and amino acids. When the pressure buildup exceeds the strength of the cellulosic cell wall, rapid popping of coffee bean occurs—a roasting event commonly known as the "second crack". In practice, these cracking phenomena are often used to evaluate the progress of roast processing of coffee beans. Accordingly, these events were chosen as the reference sampling points in the present study.

In Figure 1A, the evolution of color, moisture content, pH value, and titratable acidity of coffee samples collected at different stages of roast are presented. Figure 1B shows the corresponding changes of the same properties as a function of actual roast time.

Lightness. Color change is one of the most important modifications in coffee bean during roasting[18,19] caused by nonenzymatic browning reactions such as the Maillard reaction and caramelization.[20,21] Lightness (L^*) is often used as a

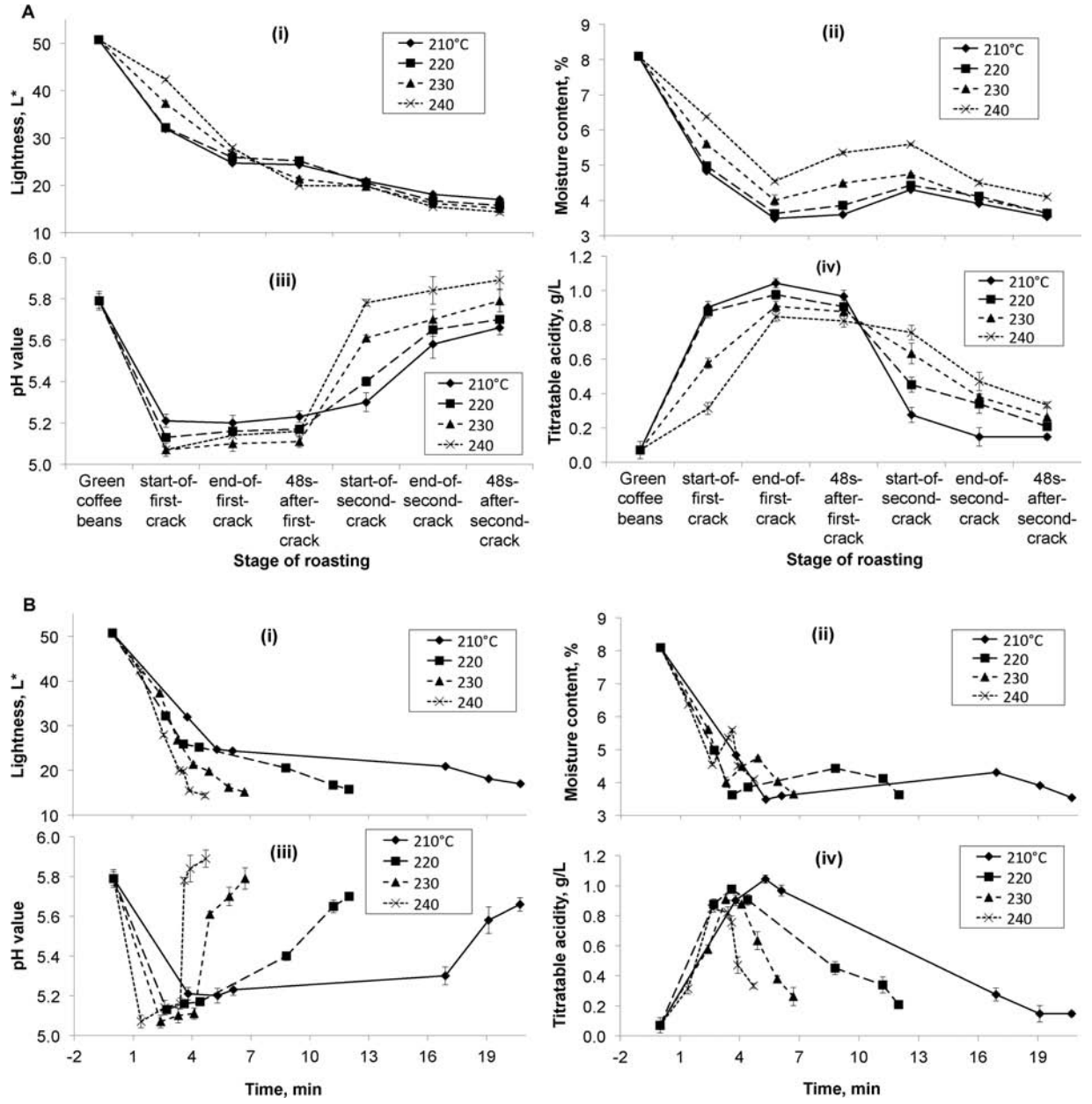

Figure 1. Changes in lightness, moisture content, pH value, and titratable acidity of coffee beans processed to different roast stages (A). The same data are plotted as a function of actual roast time (B). Roasting occurred isothermally at 210, 220, 230, and 240 °C.

measurement of the degree of roast (e.g., light, medium, and dark), which is directly related to the roasting time and temperature.[18,22,23] The L^* values at different stages of roast are summarized in panels A(i) and B(i) of Figure 1. As shown, at the end of first crack, all bean samples achieved a similar degree of roast (i.e., medium roast), with L^* values ranging between 25 and 28, regardless of the roast temperatures used. At the start of second crack, the L^* value decreased to approximately 20, as the beans attained dark roast. This result indicates that the first and second cracks corresponded to lightness of the roasted coffee, and thus these milestones could be used as one of the parameters to evaluate the degree of roast in coffee beans.

During the first phase of roasting (before the second crack), variation of lightness between beans was observed. However, when the beans were roasted to the start of second crack and onward, the lightness differences between beans reduced. This observation can be attributed to the decreased rate of lightness change as the beans were processed to darker roast. As shown in Figure 1B(i), the slopes for L^* value versus roast time plots decreased when the beans were roasted from the start of second crack onward. Moreover, the change in slope is more prominent for beans roasted at lower temperatures (e.g., 210 and 220 °C) than for those processed at higher temperature (240 °C).

Moisture Content. Panel s A(ii) and B(ii) of Figure 1 show the changes of moisture content during roasting. During the early phase of roasting (up to the end of first crack), a considerable decrease in moisture content was observed in the coffee beans. The rapid loss in moisture during the initial phase of roasting can be attributed to the vaporization of water as the temperature of the beans increased above the boiling point of water. From the end of first crack to the start of second crack, there was an increase in moisture content probably caused by

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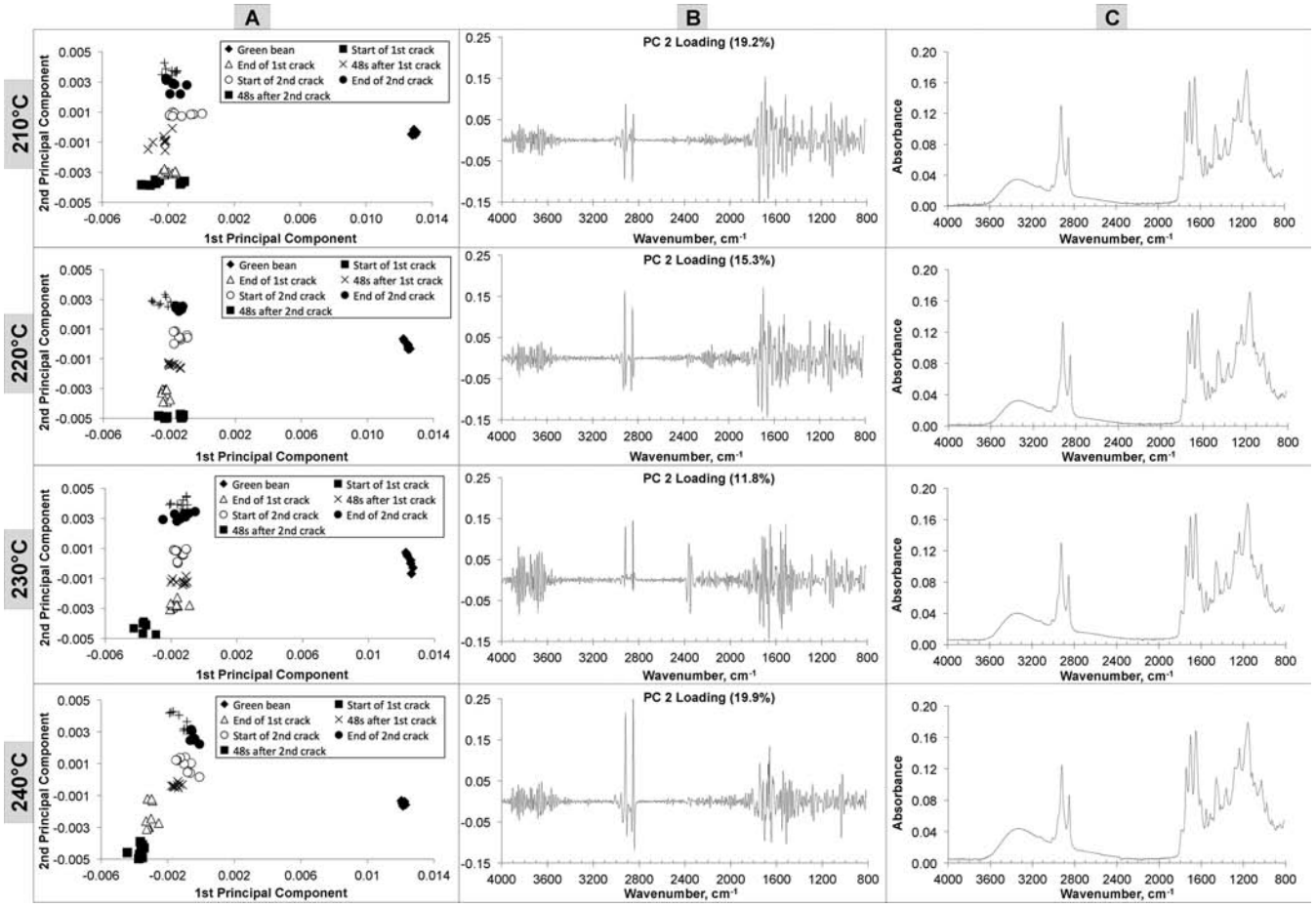

Figure 2. PCA for coffees during roasting: (A) two-factor score plots; (B) loading plots of PC2; (C) representative FTIR spectra at the start of second crack.

the generation of water as a result of pyrolytic cleavage of carbohydrates and degradation of chlorogenic acids, other organic acids, and lipids.[24,32] Beyond the start of second crack, the decreasing moisture content observed could be caused by the further rupturing of cell walls, allowing the escape of water as the roasting continued. No significant difference ($P < 0.05$) in final moisture content was observed for the final products regardless of the roaster temperature used, averaged at 3.53–3.66% dry basis. Samples roasted at 240 °C had higher moisture contents at various stages of roast as compared to those roasted at lower temperatures (Figure 1A(ii)). The observed higher moisture contents for the former may be attributed to the limiting diffusion of water from the bean to the surrounding air due to the short processing time involved.

pH Value and Titratable Acidity. The changes in pH value and titratable acidity at different stages of roast are presented in panels A(iii), A(iv), B(iii), and B(iv) of Figure 1. As shown, at all roasting temperatures investigated, a significant decrease in pH value ($P < 0.05$) was observed as the coffee beans were roasted to the start of first crack. The decrease in pH was caused by the formation of formic, acetic, glycolic, and lactic acids as the coffee beans were processed to medium roast.[25] Beyond the start of first crack, minimal changes in pH value were detected up to 48 s after first crack; thereafter, a dramatic increase in pH was observed. The rapid increase in pH value may be attributed to the destruction of organic acids formed and those that were present initially (citric acid, malic acid, and chlorogenic acids) as the coffee beans were taken to darker roasts.[25] Before the start of second crack, the pH value was lower for samples roasted at higher temperature as compared to those roasted at lower temperatures. This trend is likely caused by the faster rate of the formation of acids (the degradation of glucose, fructose, and sucrose) at higher temperatures than at lower temperatures. However, as the roasting process continued to the start of second crack, the trend was reversed, maybe because of the greater destruction of formic and acetic acids at higher temperatures.[25]

Acidity is one of the attributes commonly associated with high-quality coffees influenced by many factors such as the green beans, the roasting process, brewing water composition, and brewing method.[28] Titratable acidity and pH value are two conventions for expressing acidity. The perceived acidity of coffee is a result of proton donation of acids to receptors on the human tongue. Although many researchers observed a linear correlation between the pH value and the perceived acidity of coffee,[26,27] titratable acidity in coffee brews could also be an important indicator for correlating the coffee acidity. Recent findings from several studies on perception transduction mechanisms of acid taste suggested that undissociated forms of acid molecules could contribute to acid perception.[28,29] For example, the perceived acidity seems to cover more than 1 pH unit range despite the relatively narrow pH range exhibited by coffee Arabica brews.[28] As shown in panels A(iv) and B(iv) of Figure 1, a rapid increase of titratable acidity was observed as green coffee beans were roasted to the end of first crack (medium roast). This change in titratable acidity may be caused

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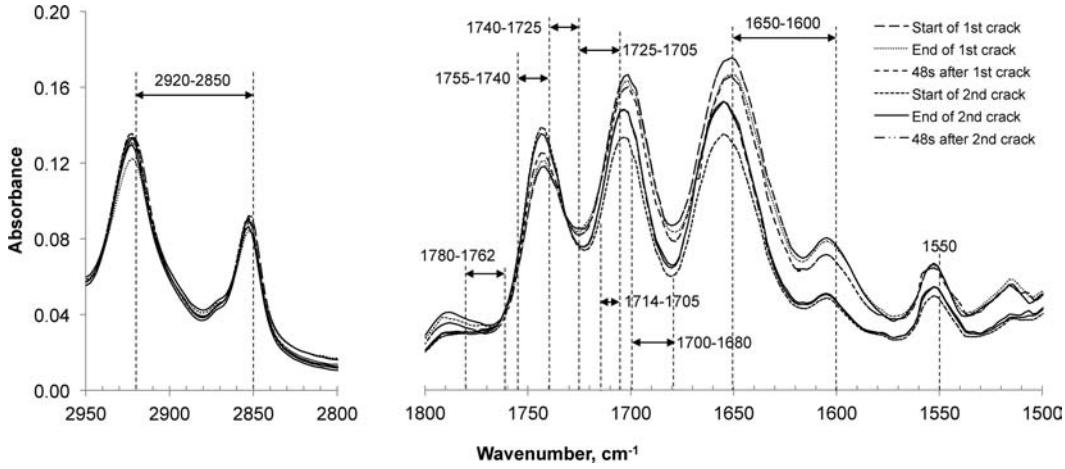

Figure 3. Expanded 2950−2800 and 1800−1500 cm^{-1} regions of the spectra of coffee roasted at 230 °C.

by the formation of total aliphatic acids to a maximum level.[30] Consistent with the pH value, further roasting from a medium to dark roast resulted in a decrease in titratable acidity, maybe because of the destruction of organic acids (e.g., citric, malic, lactic, pyruvic, and acetic acids).[31] In general, medium-roast Arabica coffee brews have a pH value ranging between 4.9 and 5.2, which is in agreement with the result from the present studies (pH 5.20, 5.16, 5.10, and 5.14 for 210, 220, 230, and 240 °C, respectively).[30] These observation suggested that by using different roast temperature and time profiles, the titratable acidity in roast coffees could be manipulated. For example, the maximum titratable acidity can be obtained by using 210 °C roast temperature before second crack started and then using 240 °C for continued roasting.

Changes in Coffee at Various Stages of Roast. Roasting is a complex process that results in substantial physical and compositional changes in coffee beans. These changes are both time and temperature dependent. In general, HTST roasting produced more soluble solids, less degradation of chlorogenic acids, lower loss of volatiles, less burnt flavor, larger volume increase, larger CO_2 desorption, and higher oil migration, as compared to the LTLT roasting process.[1,33]

In the present study, coffee beans were processed at 210, 220, 230, or 240 °C to achieve a similar degree of roast (all dark roast), based on the L^* value. Bean samples were collected at six roast stages, ground into powder, extracted in solvent, and then analyzed with FTIR spectroscopy. Typical FTIR spectra of coffee extracts are presented in Figure 2C. To determine spectral variances due to the temperature treatment, PCA was employed to reduce the dimensionality of the IR spectra to facilitate the visualization of the data set. As shown in Figure 2A, for all of the temperatures tested, the score plots displayed clusters that were separated according to different stages of roast. There is a clear trend for the clusters to move upward along the PC-2 axis as the roasting progressed through different stages. The clusters for green coffee were separated far from the other clusters, indicating that the chemical compositions within the green beans were considerably different as compared to the roasted beans, as expected.

For PC1, no separation was observed between coffees roasted to different stages when low roast temperatures were used (e.g., 210 °C). However, at higher temperature (e.g., 240 °C), the clusters had a tendency to spread along the PC1 axis as the roasting process progressed. Further analysis of PC1 loading plots (data not shown) revealed that frequencies that contribute to the spectral difference for beans roasted at 210 and 240 °C occurred at around 2920−2850 cm^{-1}, which is the absorbance range due to asymmetric and symmetric C−H stretching modes. This implies that important changes in aliphatic hydrocarbon contents had occurred during roasting, especially the lipids. This is consistent with the observation that during the roasting experiment, spots of oil were observed on the surface of beans that were roasted at 240 °C, but not on those roasted at 210 °C. The HTST process used at 240 °C might have increased the rate of oil diffusion from the bean core to the surface.[34]

To further investigate regions of spectra that contribute to the variance of samples, the loading plots for PC2 were also inspected (Figure 2B). The percent variance accounted for by PC2 is indicated on each loading plot. Regions of the spectrum with large loading score (>0.1 and < −0.1) mainly appeared at 2920, 2850, 1739, and 1660 cm^{-1}, which are due to CH_2 asymmetrical stretching of methyl groups, C—H symmetrical stretching of methyl groups, C═O stretching of polysaccharides/hemicelluloses, and C═C stretching band of lipids and fatty acids, respectively.[7,35] For coffees roasted at 220 and 230 °C, more absorbance with large loading score (>0.1 and < −0.1) was observed than for those roasted at 210 and 240 °C, especially at 1741 cm^{-1} (fatty acid esters), 1718−1707 cm^{-1} (ketones), 1697 cm^{-1} (aromatic acids), and 1514 cm^{-1} (amino groups). These compounds are important in determining the overall coffee organoleptic qualities. It has been reported that esters provide fruity aromas, whereas aldehydes/ketones are responsible for odor notes ranging from woody, cucumber, cooked fruit, and nuts. On the other hand, acids contribute to aroma attributes similar to vinegar, chocolate, and burnt caramel.[14,18,32] Silwar and Lüllmann reported that the "real" flavor of roasted coffee appeared at 220−230 °C. Beyond this point, the flavor was judged to be slightly over-roasted at 240 °C and over-roasted when processed at 250−260 °C.[36] For coffees roasted at 210 °C, some key compounds that contribute to aroma such as furans, pyrazines, Strecker aldehydes, and 2- and 3-methylbutanal might have not been fully developed.[2,36]

IR spectra for samples roasted at 230 °C were overlaid to elucidate the changes in chemical composition of the coffee extract as the roasting process progressed to various stages (Figure 3). From the literature, it is well-known that the stretching vibration of C—H bonds causes the absorbance

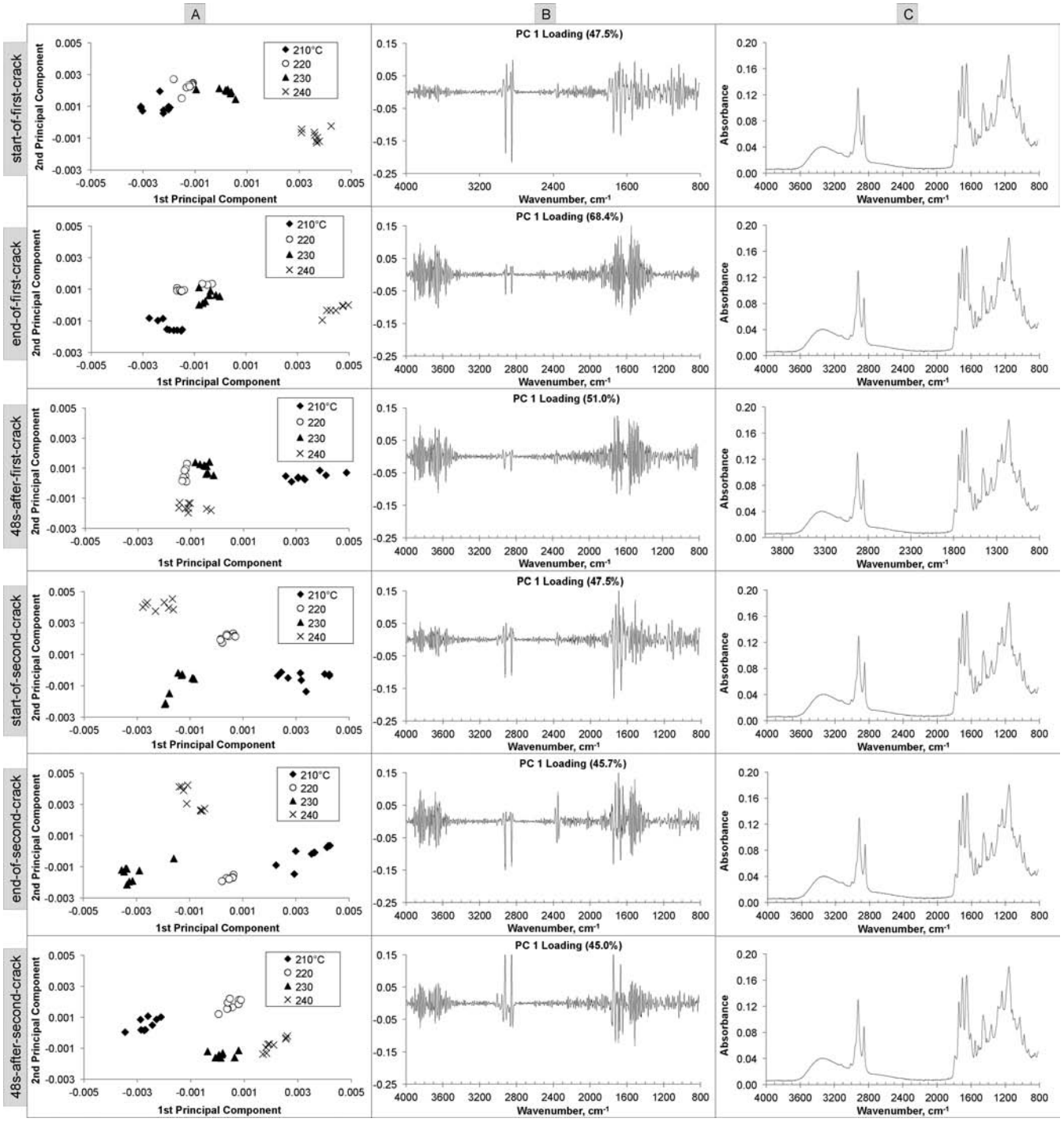

Figure 4. PCA for coffees collected at the same sampling point: (A) two-factor score plots; (B) loading plots of PC1; (C) representative FTIR spectra at 230 °C.

around 2920−2850 cm^{-1}, particularly those from lipids. Absorbance at the carbonyl region, ranging from 1780 to 1600 cm^{-1}, is attributed to the C═O stretching vibration of organic compounds, such as unsaturated ester/lactone (1780−1762 cm^{-1}), aliphatic esters (1755−1740 cm^{-1}), aldehydes (1739−1724 cm^{-1}), ketones (1725−1705 cm^{-1}), aliphatic acids (1714−1705 cm^{-1}), aromatic acids (1700−1680 cm^{-1}), and caffeine (1650−1600 cm^{-1}).[6−8,37,38] As shown in Figure 3, dynamic changes of absorbance in the carbonyl region can be seen as the coffee was roasted to different stages. For instance, the absorbance values of unsaturated ester/lactone (1780−1762 cm^{-1}) and caffeine (1650−1600 cm^{-1}) were lowered from the start of first crack to the start of second crack and then stabilized. By contrast, aliphatic esters (1755−1740 cm^{-1}) and aldehydes (1739−1724 cm^{-1}) gradually increased from the start of first crack to the start of second crack and then decreased, probably due to thermal degradation. Ketones (1725−1705 cm^{-1}), aliphatic acids (1714−1705 cm^{-1}), and aromatic acids (1700−1680 cm^{-1}) decreased initially from the start of first crack to the start of second crack and then increased as the coffee was roasted to the end of second crack. Thereafter, no detectable change was observed on further

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roasting. In comparison, spectral changes in the C—H stretching region were less obvious as compared the C═O stretching region, although the loading plots analyses (Figures 2 and 4) showed that the subtle changes in this region are important in contributing to the separation of cluster separation in PCA score plots.

Effects of Roast Temperature on Changes in Coffee. Overall, clusters for the score plots were well separated according to different roasting temperatures (Figure 4A). The separation distance between 220 and 230 °C tended to be closer during the first three stages of roast, indicating that the IR-active components are similar in the coffee beans roasted at these two temperatures. From the loading plots (Figure 4B), it is evident that coffees collected at the start of first crack were markedly different from those of the other five stages, in that the former exhibited fewer bands with large loading score, which could be caused by the fewer compounds extracted during the short processing time.[5] Loading plots for coffees collected at the end of first crack and 48 s after first crack were comparable, suggesting that the compounds present for samples roasted at different temperatures were similar. The main regions that contributed to the separation in score plots of end of first crack and 48 s after first crack were 1724−1726, 1699−1701, and 1676 cm^{-1}, due to aldehydes/ketones, aromatic acids, and lipids, respectively.[6,8,38] Other spectral regions that contributed to cluster separation were 1660 cm^{-1} due to C═C stretching band of fatty acids,[35] 1650 cm^{-1} due to amide I of protein,[11] 1550 cm^{-1} from amide II of proteins,[12] and 1514 cm^{-1} caused by N═C stretching of amino groups.[39]

At the start of second crack and the end of second crack, the separation distances between clusters increased considerably, indicating that there is an increased differentiation in the IR-active components for coffee samples processed at different temperatures. However, roasting beyond the second crack (48 s after second crack) caused the cluster separation to decrease, especially between samples processed at higher temperatures (230 and 240 °C). These results imply that the second crack is an important milestone during coffee roasting, at which the IR-active components will differ depending on the roast temperature employed.

Microstructural Analysis. SEM revealed considerable changes in the microstructure of coffee beans as they were processed to different stages during roasting (see Figure S1 in the Supporting Information). The unroasted beans displayed a relatively compact morphology as compared to the heated samples. When the beans were heated to the start of first crack, a few pores started to form. As the roasting progressed to the end of first crack, porous networks were established in the beans. Continued roasting to 48 s after first crack did not induce appreciable microstructural changes. However, when the samples were roasted to the second crack, further expansion of the pores is evident from the micrographs, as exemplified by the reduced thicknesses of the walls surrounding the pores. Furthermore, the pores were more polyhedral in shape than those appearing in earlier roast stages, suggesting that there was an increased internal pore pressure that causes the pores to compress against each other, likely due to the evolution of carbon dioxide. The carbon dioxide buildup may have also resulted in ruptured walls exhibited for some pores. Further heating to 48 s after second crack resulted in morphologies that were less uniform than the previous stages, possibly due to the artifacts caused by the disruption of brittle cell wall during sample preparation. Further examination of micrographs for samples processed to the last two roasting stages revealed the presence of droplets on the surface of the pore walls. The droplets may be attributable to the coalesced oil that formed during the extended roasting.

In general, HTST roasting processes tend to produce beans of higher porous structure in the bean cell tissues as compared to LTLT processes. The former processing condition is also known to result in higher extraction yield during solvent extraction.[40] However, at the magnification level employed during the SEM analysis and roasting conditions used, no relationship can be made between the pore size and roasting temperature. The lack of correlation could be due to the sample cutting procedure involved, which does not guarantee sectioning through the maximum diameter of pores. Density measurement will provide a more accurate assessment of sample porosity.

In summary, FTIR-ATR spectra of the ethyl acetate extract of ground coffee obtained by different time−temperature combinations contain useful information for studying the effect of roasting conditions on IR-active compounds. PCA score plots displayed clusters that were separated according to different stages of roast as well as different roasting temperatures. From this study, we can conclude that coffee beans processed to the same extent of roast (as determined from bean lightness and roast cracking milestones) by using different time−temperature roasting profiles do not necessarily possess similar physicochemical properties. Potentially, the FTIR−chemometric technique could serve as a tool for the rapid detection of process deviation in production roasters. With regard to the sensory evaluation, SIMCA models derived from FTIR spectral data will be useful to complement the cupping analysis of coffee by correlating with the sensory data, which are largely qualitative, and reducing the subjectivity of sensory evaluation.

■ ASSOCIATED CONTENT

Supporting Information

Figure S1. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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