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Mid-IR Spectroscopy as a Primary Tool in Carbon Biogeochemistry Research
Adrian Spence
Mid-infrared spectroscopy is applied as a primary technique to better understand the bio- and ultraviolet-transformation of soil microbial biomass. Soil microbial biomass constitutes as much as 50% organic carbon in soil organic matter, and therefore plays a crucial role in soil-atmospheric chemistry. In this study, the spatial distribution of microbial-derived organic structures on kaolinite and montmorillonite clay minerals was investigated.

The Application of FT-NIR and Multivariate Analysis Methods for Characterization of an Industrial Mold Purging Process
Killian Barton, Michael McAuliffe, and Liam Lewis
Near infrared (NIR) reflection spectroscopy and multivariate analysis have been used to characterize materials relating to an injection mold barrel purging process. NIR was compared to attenuated total reflectance-mid-infrared (ATR-MIR) spectral measurements to assess whether the NIR measurement was relevant to either the surface or the bulk of the samples.

Native Measurement of a Biotherapeutic without Interference from Excipients Using Microfluidic Modulation Spectroscopy
Libo Wang, Ioannis A. Papayannopoulos, Shannon Renn-Bingham, and Jeffrey Zonderman
A new infrared spectroscopy technique, microfluidic modulation spectroscopy (MMS), delivers reproducible protein characterization over close to four orders of magnitude in protein concentration (from 0.1 to 200 mg/mL). This technique characterizes samples from the earliest stages of development through to manufacture.
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Mid-IR Spectroscopy as a Primary Tool in Carbon Biogeochemistry Research

Previous studies have shown that soil microbial biomass constitutes as much as 50% organic carbon in soil organic matter, and therefore plays a crucial role in soil-atmospheric chemistry and climate. However, little is known about the fate and transformation of the magnitude of microbial components in the soil. In this study, mid-infrared (IR) spectroscopy is applied as a primary technique to better understand the bio- and ultraviolet-transformation of soil microbial biomass. The spatial distribution of microbial-derived organic structures on kaolinite and montmorillonite clay minerals was investigated to decipher which organic structures preferentially associate with the crystalline and amorphous forms of the minerals, which may be accessible to microbial heterotrophs, and which are physically protected from decomposition.

Adrian Spence

Soil organic matter (SOM) is a complex mixture of compounds with varied sources, compositions, and environmental and ecological roles. Generally, the biogeochemical characteristics of SOM represent an integrated signal of multiple processes, including degradation and its interaction with soil minerals and pedogenic oxides. Recently, the microbial contribution to SOM has been shown to be much larger than previously thought, and therefore its role in the carbon and nitrogen biogeochemical cycles may also be underestimated (1,2). Considering the huge amounts of carbon stored in soils, and the unprecedented pressure on land due to rapid population growth, climate change, land degradation, and other environmental stressors, there is an urgent need to better understand the degradation dynamics of SOM, as well as its interactions with pristine and structurally altered (weathered) clay minerals.

In soils, biological recalcitrance of organic matter (OM) can be attained through molecular structure and formation of cross linkages, but organic matter–mineral interactions have also been shown to play a crucial role in the preservation and distribution of SOM. Overall, organic matter–mineral interactions have significant implications for a number of biogeochemical processes, ranging from the formation, growth, and division of the earliest cells, to agricultural productivity and other ecosystem services, such as enhancing the ability of soils to act as a natural modulator of global climate change.
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Despite these important roles and functions, molecular-level information about the mechanisms of such interactions remains poorly understood, and even less is known about the effects of micro-scale weathering of minerals on these interactions. Therefore, these gaps in our knowledge may serve to undermine our ability to predict future carbon cycling under a warming climate, and to reconstruct paleo-environmental conditions based on organic matter-derived proxies.

In this contribution, mid-infrared (IR) spectroscopy is applied as a primary technique to better understand the bio- and ultraviolet-transformation of soil microbial biomass. Further, the spatial distribution of microbial-derived organic structures on kaolinite (1:1) and montmorillonite (2:1) clay minerals is investigated, in order to decipher which organic structures preferentially associate with the crystalline and amorphous (weathered) forms of the minerals, which may be accessible to microbial heterotrophs, and which are physically protected from decomposition. IR spectroscopy provides a rapid nondestructive means to perform molecular-level investigations on SOM and their interactions with soil minerals and pedogenic oxides. Critically, since samples are not subjected to chemical treatment, there are no side effects of secondary reactions. Additionally, the molecular composition of the sample is determined simultaneously, which simplifies and shortens the time required for spectral processing, including the quantitative determination of major macromolecular components (5).

Materials and Methods

Microbial Propagations and Conditions
A mixed heterotrophic soil biome was prepared in trypicase soy broth (TSB), supplemented with 0.5% (w/v) yeast extract. Initially, 1 g of freshly collected, homogenized agricultural soil was added to 100 mL aliquots of sterile distilled water, and dispersed by stirring. Aliquots (1 mL) of the initial soil suspension were used to prepare serial dilutions of $10^{-4}$ g soil suspension in a mixture of TSB–yeast extract and sterile distilled water (1:9). Finally, 1-mL aliquots from the serial dilutions were used to inoculate 100 mL of the undiluted growth solution, and the cultures incubated at room temperature for 72 h, with shaking at 150 rpm. Dilutions were done to prevent any possible carryover of soil particles from the initial soil suspension. After incubation, the cultures were harvested by centrifugation, and the cell pellets washed in ultrapure water and freeze-dried (6,7).

Microbial Degradation
Bio- and ultraviolet degradation of soil microbial biomass and biodegradation of kaolinite-microbial complex were conducted as described by Spence and Kelleher (8). Biodegradation was carried out under ambient conditions for 26 weeks.

Microscale Weathering of Clay Material
Microscale weathering of untreated (raw) montmorillonite (SWy-2; Source Clays Repository; The Clay Minerals Society) was carried out for 36 h, according to the protocol described by Spence and associates (9).

Adsorption Experiment
Duplicate clay-microbial adsorption experiments were carried using raw kaolinite, as well as raw and acid-treated (weathered/degraded) montmorillonite clay minerals (9).

IR Analysis
The IR spectra of initial and degraded (bio- and ultraviolet) biomass, raw kaolinite, raw and acid-treated montmoril-
lonite, and their related microbial complexes were acquired on a Bruker Tensor Series FT-IR spectrometer, using the KBr pressed disk technique. Thoroughly dried samples (~1 mg) were homogenized in 100 mg of spectroscopic-grade KBr (Sigma). Background KBr spectra were obtained and spectra ratioed to the background. Spectra were recorded by accumulating 256 scans in the 4000 to 400 cm⁻¹ mid-IR spectral range in the absorbance mode, with a resolution of 4 cm⁻¹. To minimize complications arising from unavoidable spectral shifts, baseline correction was applied to all spectra using the automatic baseline correction method. In addition to the use of thoroughly dried samples, it was essential to analyze the disk-like pellets immediately after preparation. This was done to minimize the adsorption of atmospheric water by the hygroscopic KBr, hence reducing the possibility of chemical exchange between the clay and KBr, which could result in a change of the crystalline structure of the mineral (10). Further, rapid analysis of the pellets was important to eliminate the suppression of key signals from the samples by KBr-adsorbed water.

**Results and Discussion**

Figure 1 presents the IR spectra of fresh microbial biomass (a), and that degraded under ambient (b) and ultraviolet (c) conditions. General peak assignments are applicable to all spectra, and the characteristic functionalities of major biochemical
components are summarized in Table I. After degradation has occurred, major structural changes are observed, particularly the enrichment of polymethylenic-C \((\text{CH}_2)_n\) in long-chained aliphatic structures (\(\text{CH}_x\) stretching bands at 3000 cm\(^{-1}\) to 2700 cm\(^{-1}\)), indicating that they are selectively preserved as part of the stable carbon pool. Signals from other biochemical components (proteins and carbohydrates; see Table I) are attenuated, indicating that they have degraded relative to aliphatics. Overall, there is no distinction between bio- and photodegradation products. Bio- and photodegradation are primary decomposition processes involved in organic matter transformation in the terrestrial environment (8).

The superimposed spectra of raw and acid-treated montmorillonite are presented in Figure 2. A band at 3626 cm\(^{-1}\) denotes \(v(\text{OH})\) of structural OH groups coordinated to octahedral cations (\(\text{Al–O(OH)}\)); Al-Al-OH bending (917 cm\(^{-1}\)), Al-Fe-OH (881 cm\(^{-1}\)), and Al-Mg-OH (844 cm\(^{-1}\)), suggesting that octahedral Al\(^{3+}\) is partially substituted by Fe\(^{2+}\) or Mg\(^{2+}\) ions (11). Two shoulders near 881 cm\(^{-1}\) and 844 cm\(^{-1}\) suggest a relatively low concentrations of Fe\(^{2+}\) and Mg\(^{2+}\) ions, respectively. After acid-treatment, the \(v(\text{OH})\) band (3626 cm\(^{-1}\)), the –OH bending vibrations near 917, 881, and 844 cm\(^{-1}\), and \(\delta(\text{Si–O–Al})\) at 526 cm\(^{-1}\) (12) are no longer observed, demonstrating a depletion in the content of Al\(^{3+}\) and substituted Mg\(^{2+}\) and Fe\(^{2+}\) cations in the octahedral sheet. This is supported by the attenuation of spectral hydration peaks in the –OH stretching (3419 cm\(^{-1}\))
and \(v_2\)(H\(_2\)O) bending regions 1650–1623 cm\(^{-1}\), and an incremental shift in the position of the \(v_2\)(H\(_2\)O) peak from 1623 to 1620 cm\(^{-1}\). A shoulder near 3225 cm\(^{-1}\), indicating an overtone (2\(v_2\)) of bending mode of cation hydration water in the raw mineral, is not observed in the acid-treated clay. Microscale weathering of the tetrahedral (Si environment) is also observed, and is characterized by changes in both the shape and position of the Si–O stretching band (1047 cm\(^{-1}\), now appearing at 1095 cm\(^{-1}\), and the enrichment of the asymmetric band 790 cm\(^{-1}\). This would suggest that acid hydrolysis (a key process in soil formation) has transformed the crystalline structure of montmorillonite to amorphous silica. All remaining bands (1095, 790, and 463 cm\(^{-1}\) [12]) in the acid-treated spectrum are synonymous with Si–O vibrations.

Figure 3 illustrates the interactions of microbial biomass with raw and weathered montmorillonite. Microbial interactions with raw montmorillonite is dominated by signals from aliphatic and proteinaceous components (Figure 3a; see Table I for full assignment of spectral peaks). The results also clearly demonstrate that only limited quantities of microbial-derived components (primarily proteins [1640 cm\(^{-1}\)]) are adsorbed to the weathered structure. This would suggest that octahedral and interlayer cations present in a crystalline structure play important roles in the adsorption process.

The IR spectra of raw kaolinite (a), kaolinite–microbial complex (b), and the lat-
ter sample degraded under ambient conditions for 26 weeks (c) are presented in Figure 4. Raw kaolinite exhibits four characteristic peaks assigned to $\nu$(OH) stretching vibrations of surface OH groups (3652, 3671, and 3694 cm$^{-1}$), and $\nu$(OH) vibrations of inner OH groups (3620 cm$^{-1}$). The $\nu$(Si–O) is represented by three strong, well-resolved bands in the 1100–1000 cm$^{-1}$ region. Bands at 936 and 913 cm$^{-1}$ represent OH-bending vibrations of the outer and inner surface OH groups of the mineral, respectively. Additional bands near 701 and 755 cm$^{-1}$ are associated with the surface OH groups. Bands due to $\delta$(Al-Fe-OH) at 865–875 cm$^{-1}$ and stretching at 3607 cm$^{-1}$ are typical of Fe-bearing kaolinites, while the absorption bands due to Si–O–Al and Si–O–Si are observed at 544 and 473 cm$^{-1}$, respectively (13). After interactions have occurred, the $\nu$(OH), $\delta$(OH), $\nu$(SiO), and the $\delta$(SiO) bands are now attenuated (Figure 4b and c). Further, the $\nu$(OH) band at 3671 cm$^{-1}$ is no longer observed, suggesting that hydrogen bonding with microbial-derived components has occurred.

Conversely, the bands at 3621, 3652, and 3694 cm$^{-1}$ remained unchanged, indicating there was no intercalation of the mineral. Signals from $\nu$(OH) in phenolic and carboxylic groups and $\nu$(N-H) of amides from major biochemical groups appear as a strong broad at 3408–3300 cm$^{-1}$.
cm$^{-1}$. Microbial–mineral interactions are further evidenced by peaks at 2929 and 2856 cm$^{-1}$ assigned to $\nu_{as}(\text{CH}_2)$ and $\nu(\text{CH}_2)$ from CH$_2$ methylene groups, respectively. When coupled with a weak shoulder at 2960 cm$^{-1}$ ($\nu_{as}[\text{C–H}]$ of methyl groups), this would suggest a contribution from polymethylene-C ($[\text{C–H}_2]_n$) with a low degree of branching in the interaction process (14). Contributions from other biochemical components with roles in the interaction process, including bands at 1657 cm$^{-1}$ attributed to aromatic $\nu(\text{C=C})$ bonds, $\nu(\text{O–C}=\text{O})$ of metal-coordinate carboxylates, H-bonded $\nu(\text{C}=\text{O})$ of amides, and amide I band in peptides and proteins. Absorption peaks of moderate to low intensity at 1545, 1425, 1394–1380, and 1235 cm$^{-1}$ denotes $\delta(\text{N–H})$ of amide II of proteins; $\delta(\text{O–H})$ of carboxylic acids, the CO$_2$ stretch of carboxylates and the aliphatic CH$_2$ group of alkanes; $\delta$(CH$_2$) and $\delta$(CH$_3$), phenolic $\nu(\text{C–O})$ or $\nu_{sym}(\text{OCO})$ of coordinated carboxylate groups; and $\nu(\text{C–O})$ of carboxylic acid and the $\nu(\text{C–N})$ of amides (amide III), respectively (14). A weak shoulder at 1099–1095 cm$^{-1}$ corresponds to the C–O–O and C–O ring vibration of carbohydrates, most likely from bacterial cell wall components such as peptidoglycan. Note, these figures remain largely unchanged following degradation of the clay-microbial complex, suggesting that microbial-derived biomolecules are physically protected.

**Conclusions**

In this study, mid-infrared (IR) spectros copy is applied as a primary technique to better understand the bio- and ultraviolet-

### Table I: Positions and assignments of adsorption bands observed in the mid-iR spectrum of soil microbial biomass (in cm$^{-1}$).

<table>
<thead>
<tr>
<th>Observed Wavenumber (cm$^{-1}$)</th>
<th>Band Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>3308</td>
<td>$\nu(\text{OH})$ of hydroxyl groups</td>
</tr>
<tr>
<td>3261</td>
<td>$\nu(\text{N–H})$ of amide A in proteins; $\nu_{as}(\text{O–H})$ of H-bonded phenolic groups</td>
</tr>
<tr>
<td>2961</td>
<td>$\nu_{as}(\text{C–H})$ of CH$_3$ in aliphatic structures</td>
</tr>
<tr>
<td>2929</td>
<td>$\nu_{as}(\text{C–H})$ of CH$_2$ in aliphatic structures</td>
</tr>
<tr>
<td>2852</td>
<td>$\nu(\text{C–H})$ of CH$_2$ in aliphatic structures</td>
</tr>
<tr>
<td>1650–1640</td>
<td>$\nu(\text{C}=\text{C})$ in aromatics, $\nu(\text{C}=\text{O})$ in amide I</td>
</tr>
<tr>
<td>1546</td>
<td>$\delta(\text{CH}_2)$, $\delta(\text{CH}_3)$, $\nu(\text{C–O})$, $\delta(\text{N–H})$ in plane</td>
</tr>
<tr>
<td>1460</td>
<td>$\delta(\text{C–H})$ of CH$_2$ and C(CH$_3$)$_2$ in aliphatic structures</td>
</tr>
<tr>
<td>1401</td>
<td>$\delta(\text{C–H})$ of CH$_2$ and CH$_3$ in aliphatic structures; $\nu(\text{C}=\text{O})$ of COO</td>
</tr>
<tr>
<td>1242</td>
<td>$\nu(\text{C–O})$ of COO; $\nu(\text{C–N})$ of amide III</td>
</tr>
<tr>
<td>1085</td>
<td>$\nu(\text{C–C})$, $\nu(\text{C–O})$, $\nu(\text{C–O–C})$ predominantly from ring vibrations of carbohydrates; $\nu(P–O)$ of nucleic acids</td>
</tr>
<tr>
<td>990–500</td>
<td>$\delta(\equiv\text{C–H})$ or aromatic</td>
</tr>
</tbody>
</table>

$\nu = $ symmetric stretching; $\nu_{as} = $ asymmetric stretching; $\delta = $ bending.
transformation of soil microbial biomass. The spatial distribution of microbial-derived organic structures on kaolinite and montmorillonite clay minerals was investigated to decipher which organic structures preferentially associate with the crystalline and amorphous forms of the minerals, which may be accessible to microbial heterotrophs, and which are physically protected from decomposition.

Bio- and photodegradation produced indistinguishable results, and are both characterized by considerable enrichment in aliphatic components, presumably polymethylenic-C ([C-H₂]ₙ), and a concomitant decrease in the relative concentrations of carbohydrate and protein structures in a clay-free environment. Results also indicate that microbial-derived organic matter (OM), primarily aliphatic components, adsorbed mainly to the external surfaces of the minerals, but also to the interlamellar region of montmorillonite. Similarly, aliphatic structures appear to be dominant after degradation of OM-mineral complexes had occurred. After chemical weathering of montmorillonite, the adsorption of microbial-derived components (in particular lipids) to the amorphous form of the mineral appears to have decreased. Based on this behavior, an immediate conclusion is that spatial co-variation of microbial-derive OM with octahedral cations in the minerals may be a primary mechanism of interaction.

References


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TGA-FTIR as a Tool for Investigating Plastic Failures

Frank Wasacz, Thermo Fisher Scientific

Investigating the failure of plastic materials often requires using a variety of analytical tools to identify the mechanism and the cause of the failure. Of the many tools available, FTIR is often one of the first tools employed for root cause analysis. FTIR can offer valuable insights into many of the causes of failure from incorrect materials used in the formulation to the identification of chemical agents in contact with the failed part.

A broken cell phone case after microscopic investigation showed surface fractures consistent with environmental stress cracking due to tensile stress and exposure to an incompatible chemical agent.

Experimental Conditions
Spectra were collected in the area of the fracture utilizing the built-in diamond ATR module of a Thermo Scientific Nicolet™ iS50 FTIR spectrometer. A small amount material from the failed area was analyzed by TGA-FTIR from 25 °C to 500 °C utilizing the Nicolet iS50 TGA module.

Results
The analysis of the ATR spectrum confirmed the correct material composition but also showed a spectral feature not attributed to the plastic. A spectrum extracted from the weight loss event at 400 °C was analyzed using OMNIC™ Spectra Multi-Component Search. Two of the components were expected breakdown components of the polymer while the third was a methyl ester. The most likely scenario that caused the failure was that the case was often in contact with hand cream and some of the excess ester in that cream penetrated into the polymer matrix, resulting in the pre-disposition for cracking.

Conclusions
The combination of ATR and TGA-IR is a powerful tool in the identification of the mechanism and cause of failures in plastic components.

Figure 1: Spectrum A extracted from TGA-IR data set collected from material in the area of crack. Spectra B, C, and E are library matches from an OMNIC Spectra Multi-Component Search identifying three components. Spectra B and C are both expected breakdown components from the polymer while the material identified in spectrum C of methyl ester was not.
The Application of FT-NIR and Multivariate Analysis Methods for Characterization of an Industrial Mold Purging Process

A combination of near infrared (NIR) reflection spectroscopy and multivariate analysis has been used to characterize materials relating to an injection mold barrel purging process. Reference spectra from unprocessed mold and purge materials have been compared with samples prepared after purging to estimate the content of residues in samples after a series of purging steps. Based on the results of principal component analysis and multivariate curve resolution, injection molded samples compare best with reference nylon after two purges of the barrel. Comparison of analysis of the results from the NIR and attenuated total reflectance-mid-infrared (ATR-MIR) spectral measurement was carried out to assess whether the NIR measurement was relevant to either the surface or the bulk of the hub samples.

Killian Barton, Michael McAuliffe, and Liam Lewis

Nylon hubs, used in the construction of neurovascular catheters, may be manufactured using injection molding. After repeated use, nylon residues build up in the barrel, and these must be removed to prevent contamination. This is accomplished by using a purging material that is fed through the injection mold barrel, removing residues as it is passed through. In doing so, the barrel becomes contaminated temporarily by the purge material, and this is removed by running several blanks of the material that is normally injection molded using the purged barrel (1).

The industrial client for whom we carried out this analysis has switched from one purging material to another. They wished to determine the point at which the hubs are clear of residues associated with the barrel and the purge material. Determination of the point at which the barrel residues and purging materials are no longer present, as contamination in the molded pieces is often estimated using visual inspection alone. However,
this method has an inherent uncertainty. We present a method based on the combination of near-infrared (NIR) spectroscopy and multivariate analysis techniques that allows for the clear identification of the point at which no further purging is necessary.

NIR spectroscopy is an established technique used for analyzing polymers. Absorption of NIR light by a polymer sample may occur by interaction with vibrational overtones and sum tones of chemical functional groups such as N-H, C-H, and O-H. These functional groups are common to a range of polymers, and contribute to a given polymer’s spectral fingerprint. The spectrum may be used to discriminate between materials and to measure concentrations of mixtures.

Measurement of the NIR spectrum is non-destructive, and sample preparation is generally minimal or unnecessary (2). Principal component analysis (PCA) is a multivariate analysis technique that is sensitive to changes in process conditions. The scores calculated from PCA of a data set allow for visualization of the relationships between the samples in a set. Clusters, patterns, and trends are revealed, and these are interpreted based on the context of and prior knowledge relating to the sample set (3). Multivariate curve resolution (MCR) is a complementary multivariate analysis technique to PCA. It differs from PCA in that the information used in the analysis is not abstract. Loadings describing components calculated using PCA are abstract solutions, whereas those
calculated using MCR or inputted into an MCR model are related to the actual physical characteristics of the sample. It is often used in the spectroscopic analysis of mixtures (4).

**Materials and Methods**

Nylon pellets, polystyrene pellets, and sample hubs were supplied by Stryker Neurovascular. Sample hubs or “blanks” were molded and collected after purging of the mold barrel. Blanks were collected from each of the five moldings after the first purge. Blanks collected after the second and third purges were taken from the first and fifth moldings.

Optical imaging of the hubs was carried out using an Ash Inspex inspection camera and a Leica M165 inspection microscope. NIR spectra were recorded using a Perkin Elmer Spotlight 400 fitted with a NIRA reflection NIR accessory. Each spectrum was accumulated from 32 scans at 16 cm\(^{-1}\) resolution. ATR-MIR spectra were recorded using a Perkin Elmer UATR accessory. Each spectrum was averaged from 16 scans at a resolution of 1 cm\(^{-1}\).

Multivariate analysis of spectral data was carried out using Camo Unscrambler software. Principal component analysis (PCA) was calculated using a

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**Figure 2:** Imaging of the hub area measured using the NIR spectrometer. The sampling area of the NIR measurement is shown on sample (e); (a) through (f) show different conditions (as indicated for each inset figure).
singular value decomposition (SVD) algorithm. PCA was calculated using the first derivative of the NIR spectra. The derivative spectra were calculated using a Savitzky-Golay algorithm with a second order polynomial. Derivative spectra were processed using standard normal variate (SNV) normalization. Multivariate curve resolution (MCR) was calculated using an alternating least squares algorithm with a maximum number of 50 iterations. The constraints of non-negativity and closure were applied to the MCR model. The spectra of the reference polystyrene and nylon were used as pure components in an “initial guess” model. MCR was performed on spectra processed using SNV normalization.

Results and Discussion
Initial assessment of the hubs was carried out visually. In the case of the blanks prepared after the first purge, purge residue carried over from the process is visible as cloudy material distributed heterogeneously through the hub body. The fourth and fifth blanks are included in Figure 1 as a reference to visible hub contamination after purging. The first and fifth blanks prepared after two subsequent purges showed no visible signs of purge residue carry over. With visual examination alone, it is not certain whether the polystyrene used to purge the injection mold barrel is completely absent from these sets of blanks. For example, the cloudy material observed for the blanks prepared after the first purge may be attributed to the purge polystyrene, residues from the barrel or a combination of both.

NIR measurements were made on the blank hubs, the purge material and the nylon pellets. Figure 2 shows the part of the hub at which the NIR measurements

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Figure 3: NIR spectra (as absorbance vs. wavenumbers) for reference materials nylon, polystyrene, and P1 B5. The spectra presented here are offset, and have been processed using standard normal variate (SNV) correction.
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Figure 4: ATR-MIR spectra (as absorbance vs. wavenumbers) for reference materials nylon, polystyrene, and P1 B5. The spectra presented here are stacked, and have been processed using standard normal variate (SNV) correction.

Figure 5: Principal component analysis (PCA) of “blank” hubs after purging and reference materials (nylon and polystyrene).
was made. Figure 3 shows examples of the NIR absorption spectra recorded from the hub samples. The reference spectra for the purge and hub materials have distinct peaks associated with their chemical composition that allow them to be distinguished from one another. The NIR spectrum of polystyrene exhibits absorption peaks related to the first and second overtones of the aromatic C-H stretch vibration at 5945 cm⁻¹ and 8743 cm⁻¹, respectively (5). The NIR spectrum of nylon exhibits absorption peaks assigned to the amide N-H group found at 4880 cm⁻¹ and 6752 cm⁻¹. C-H absorption peaks relating to the first and second overtones of the stretching absorption are observed in the nylon spectrum at 5786 cm⁻¹ and 8263 cm⁻¹, respectively. In the polystyrene spectrum, these peaks are observed at 5792 cm⁻¹ and 8268 cm⁻¹, respectively (6).

PCA of the spectral data is shown in Figure 5. The blank hubs from the first five runs after the first purge show the largest scatter in the plot. The first blank after the first purge groups closest with the score calculated for the purge material. Scores of subsequent samples converge toward that of the nylon reference material. Of the blank samples, the fifth hub from the second purge shows the closest score to that of the nylon reference. This is more clearly presented in Figure 6, where the scores for each sample in the first principal component are presented. One can see there that 94% of the variance in \( x \) is explained by the first principal component in the PCA model, and it is interpreted as the component that differentiates between the purge residue and the nylon. This result indicates that the fifth blank of the second purge has the least post-purge contamination relative to the others measured. In the case of both the second and third purges, the fifth blank grouped more closely with the nylon reference spectrum than the first, indicating that the

![Figure 6: Scores for the hub test samples and materials from the first principal component.](image)
fifth blank has less contamination than the first for each purge.

MCR analysis of the spectral data was carried out in order to compare the results with those obtained using the PCA (Figure 7). The trend observed agrees with those found in the results of the PCA. The blanks prepared after the first purge show the largest amount of purge material present relative to samples from subsequent purges. For the second and third purges, the first blank after purging shows a larger degree of purge residue contamination relative to the fifth.

It is assumed that the NIR beam penetrates into the bulk of the hub during measurement and, in this manner, reveals bulk properties of the sample. Evidence supporting the validity of this assumption was revealed by performing PCA on ATR spectra of the blank hub samples (Figure 4). Figure 8 shows the scores plot calculated for ATR spectra recorded from the surface of each hub blank. The first principal component (PC 1) describes the difference between the spectral response of the nylon and the polystyrene reference materials. It accounts for 98% of the variance in the data. The first hub of the first purge is the only hub that shows any significant trending toward the polystyrene region of the scores plot. The spectral data of the remaining hubs score similar to the nylon reference for this component. This indicates that the NIR measurements have a better sensitivity to the composition of the bulk of the polymer sample than the ATR measurement.

Conclusions
A combination of NIR and multivariate analysis techniques has allowed for a detailed characterization of changes in
material composition during a purging process. The information revealed allows for a reliable identification of the point at which the purging process is complete, and no further purging is necessary. This information is useful in the context of process management for routine implementation and efficiency; in particular, in an industrial context where unnecessary process steps can lead to large waste in the context of annual accumulation. The technique described is non-destructive, and the results are relevant to the bulk composition of the materials of interest.

**References**


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**Figure 8:** Scores plot calculated for the ATR spectra recorded at the surface of selected hub samples from the first and second principal components.
Native Measurement of a Biotherapeutic without Interference from Excipients Using Microfluidic Modulation Spectroscopy

A defining requirement in biopharmaceutical development, formulation, and manufacturing is to elucidate and maintain the structure of the drug entity. That work relies on relevant protein characterization, a task complicated by how samples evolve through the drug development lifecycle. The requirement to change techniques to cope with progressively more concentrated, and complex, multicomponent formulations is a major issue when it comes to comparing datasets and safeguarding structural parity. In this article, we discuss a new infrared spectroscopy technique, microfluidic modulation spectroscopy (MMS), that delivers reproducible protein characterization over close to four orders of magnitude in protein concentration, from 0.1 to 200 mg/mL. This technique can transition with the sample from the earliest stages of development through to manufacture, and present experimental data demonstrating the concentration-independent data that can be generated.

Libo Wang, Ioannis A. Papayannopoulos, Shannon Renn-Bingham, and Jeffrey Zonderman

Elucidation of the structure–function relationships that define the clinical efficacy of therapeutic biologics such as monoclonal antibodies (mAbs) makes protein characterization critical to their development. Though the primary structure of proteins is relatively stable, higher order structure (HOS)—secondary, tertiary and quaternary—is labile, susceptible to change triggered by chemical or thermal stress. Therefore, delivery of a drug entity to the patient with its structure intact, unchanged by manufacture or storage, relies on detailed protein characterization across the biopharmaceutical lifecycle. This requirement creates the need for an analytical technique that can characterize structure reproducibly across a broad range of concentrations, and that remains viable and reliable in the presence of excipients and additives.
Secondary structure develops from interactions between hydrogen bond donor and acceptor residues associated with repeating units in the primary peptide chains of the protein, and is typically characterized by Fourier transform infrared (FT-IR) spectroscopy and far ultraviolet circular dichroism (far-UV CD). The most common element of secondary structure is the α-helix, but β-sheet structures are also relatively prevalent, particularly within mAbs, which is one of the most widely used classes of therapeutic proteins. β-sheet structures consist of β-strands and stretched sections of peptide chain, linked via hydrogen bonds (1).

With FT-IR, information relating to secondary structure is extracted from absorption spectra spanning the amide I band, resulting from the C=O stretch vibrations of peptide linkages along the protein backbone. These modes are highly sensitive to changes in secondary structure (2), with features of the amide I band correlated robustly with specific secondary structure elements; sensitivity to β-sheet structure is a particular benefit (3,4) of using the mid-IR. However, from a practical perspective, conventional FT-IR technology has some limitations, including the relatively high concentration required for measurement, optimally in the range of 10–150 mg/mL, which makes the technique less viable for the earlier stages of drug development, when sample concentration is typically low. In addition, FT-IR tends to exhibit background drift and poor repeatability, due to the complexities of water vapor subtraction and referencing.

The technique of circular dichroism (CD) characterizes samples on the basis of the differential absorption of left-handed and right-handed circularly polarized light that occurs when a molecule contains one or more chiral chromophores. Such chromophores include the peptide bonds associated with secondary structure, which give rise to a CD signal in the far-UV region (180–240 nm) that can be analyzed to determine the relative quantities of major secondary structural elements (5). Far-UV CD is well-suited to more dilute, simple solutions, operating at a relative low concentration of 0.2 mg/mL, for example, when using a cell of 1-mm pathlength. However, commercial products are typically formulated at higher concentrations, and the presence of chiral molecules in more complex formulations is also problematic. Typically, a dialysis procedure must be performed to remove excipients in the formulation buffer that interfere with CD signals. Phosphate buff-

**Figure 1:** With MMS, rapid modulation with a matched reference stream produces highly sensitive differential IR scans, eliminating any potential interference from formulation excipients and additives; a tunable, cascade laser and optimized optical set-up enable measurement across a very wide concentration range.
Buffer subtraction and concentration normalization produces absolute absorbance spectra for the mAb that can be used for detailed structural analysis.

Switching from far-UV CD to FT-IR as samples become more complex and concentrated, through formulation and into commercial manufacture, is one way to address the limitations of these techniques. However, this introduces the complicating factor of determining whether any conflicting observations are scientifically relevant, or simply an artifact of measurement or data analysis. An alternative strategy is to dilute samples...
to extend the application of far-UV CD. This is problematic from the perspective of data relevance, given that proteins are highly sensitive to their environment. The limitations of both of these strategies creates a need for innovative spectroscopy platforms for the direct measurement of native proteins.

An important step forward in this respect is the development of microfluidic modulation spectroscopy (MMS), a technique that harnesses the advantages of IR

**Figure 4:** One way to highlight difference, or conversely confirming similarity, between samples is to analyze the second derivative plots, as shown.

**Figure 5:** Higher order structure (HOS) analysis determines the quantity of specific elements of secondary structure present in the mAb, including beta sheet, beta turn, unordered structure, and alpha helix.
IR Technology for Today’s Spectroscopists  August 2019

spectroscopy for robust measurement across a very wide dynamic range, with minimal interference from formulation excipients and additives (see Figure 1). In the MMS system, the sample solution and a matching reference stream are rapidly modulated (1–5 Hz) across the laser path to produce differential absorbance spectra via a process of continuous autoreferencing. The result is nearly drift-free scans background compensated to the buffer of interest, and of superior quality to those produced via conventional FT-IR. Concentration limitations are also directly addressed through the use of a tunable mid-IR quantum cascade laser that gen-

![Figure 6: The protein concentration plot for the PBS dilution series showing (a) a very high degree of linearity; and (b) absolute absorbance spectra that are very closely matched, indicating a high degree of structural similarity in the samples.](image1)

![Figure 7: The HOS of the mAb in the PBS dilution series is closely similar to the formulation dilution series with beta sheet and beta turn the predominant structural elements.](image2)
erates an optical beam around 100 times brighter than that used in a typical FT-IR. Run in continuous wave mode to generate a very accurate, low noise beam, this laser, in combination with a fully optimized optical configuration, delivers highly repeatable, high sensitivity measurement over a concentration range from 0.1–200 mg/mL. The laser also enables the use of simple detectors with no requirement for nitrogen cooling.

MMS effectively shapes IR spectroscopy into an efficient tool for protein characterization from early drug development through to manufacture. The following experimental study of a mAb in clinical development demonstrates the capabilities of MMS for the measurement of protein samples in the presence of different buffers and across a very broad concentration range.

**Experimental**

Dilution series were prepared from a mAb stock sample of concentration of 157 mg/mL made up in a formulation buffer. A first dilution series was prepared by mixing volumes of the stock solution with the formulation buffer to create a samples series covering a concentration range from 1 to 80 mg/mL (specifically 1, 5, 10, 20, 40, and 80 mg/mL). A second dilution series was prepared in a strictly analogous way, and covering the same concentration range, by mixing samples of the stock solution with a PBS buffer at pH 7.4. In addition, a series of samples of formulation buffer diluted in PBS buffer were prepared as matching buffers for the PBS dilution

**Figure 8:** Combining second derivative spectra for all of the samples in both buffers confirms the exemplary similarity of secondary structure across both dilution series.

<table>
<thead>
<tr>
<th>Conc. (mg/mL)</th>
<th>Similarity (%) of replicates</th>
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<tbody>
<tr>
<td>1</td>
<td>98.19 98.54</td>
</tr>
<tr>
<td>5*</td>
<td>99.90 99.90</td>
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<tr>
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<td>40</td>
<td>99.61 99.62</td>
</tr>
<tr>
<td>80</td>
<td>99.55 ---</td>
</tr>
</tbody>
</table>

*Similarity (%) was calculated by comparing to the mean of 5 mg/mL sample replicates.
series. In MMS measurements, the sample is modulated with a relevant buffer, so there is a need for a series of PBS diluted formulation buffer samples matched to the PBS dilution series samples.

All samples from both dilution series were analyzed by MMS (AQS3pro, RedShiftBio), generating differential absorbance spectra for each sample. All samples were tested at a modulation rate of 1 Hz. The instrument automatically determines the back pressure required for optimal fluid flow through the flow cell, to enhance data quality. For samples in the 1–40 mg/mL concentration range, the applied back-pressure was 5 psi and duplicate measurements were carried out for each sample. For the most concentrated 80 mg/mL samples, which were more viscous, higher back pressures were automatically determined (25 psi for the sample in the formulation buffer, and 10 psi for the sample in the PBS buffer). One measurement was carried out for both of these samples. The measurements made were subject to comprehensive data analysis using the instruments integral software package (AQS3delta, RedShiftBio).

**Results and Discussion**

**Analyzing MMS Data**

Analysis of the dataset for the first dilution series highlights key features of the data generated by MMS, and how it can be processed to reveal information about secondary structure, and to compare samples.

Figure 2a shows the differential absorbance spectra produced for the first dilution series; the replicate measurements indicate the high repeatability and accuracy. A plot of the maximum diffAU value as a function of concentration (see Figure 2b) shows excellent linearity with an $R^2$ value of 0.9997, illustrating the potential of the technique for protein quantification. Samples of unknown concentration can be accurately quantified using this plot.

With the buffer subtracted and concentration normalized, the absolute absorp-
tion spectra for each of the samples can be compared directly (see Figure 3). The high degree of overlap in these spectra indicate that the secondary structure of the protein is essentially unchanged by the dilution process across the full concentration range, which is not the case for all proteins.

One way to accentuate structural differences between samples is to generate second-derivative spectra from absolute absorbance spectra, as a function of wavenumber. This data processing step produces the traces shown in Figure 4, which provide further evidence of the close similarity of the samples, particularly those in the 5 to 80 mg/mL range.

Protein similarity can also be directly compared via area of overlap (AO) plots (data not shown) which quantify the extent to which the spectra for two samples are similar (see Table I). Using the average AO plot for the two 5 mg/mL samples as the reference for comparison, this analysis indicates that the 1 mg/mL samples have a similarity of 98.2 to 98.5%, while all the remaining samples, across the remainder of the concentration range, have a similarity in the range 99.6 to 99.9%.

Finally, because absorption at specific wavenumbers is well-correlated with specific elements of secondary structure, the relative quantities of the different elements can be determined (see Figure 5). The secondary structure of the mAb is shown to consist predominantly of beta sheets (~60%) and beta turns (~30%), with a much smaller fraction of unordered structure and minimal levels of alpha-helix. Levels of each element are extremely consistent across each of the samples.

Analyzing the PBS Dilution Series
The data processing steps illustrated in the preceding stage were applied to elucidate the impact of diluting the mAb stock with PBS buffer. Figure 6 shows the protein concentration plot for the PBS dilution series alongside the absolute absorbance spectra.

These data show that dilution in the PBS has a closely comparable impact to dilution in the formulation buffer. The plot of maximum diffAU against protein concentration, for example, shows similar, comparably high linearity, exhibiting an \( R^2 \) value of 0.9998. The absolute absorbance spectra indicate that the structure of the protein is closely similar across the full concentration range, as confirmed by the HOS analysis (see Figure 7).

These results are directly useful when it comes to comparing measurements with far-UV CD with MMS, or, indeed, FT-IR. The mAb stock solution was supplied at a concentration of 157 mg/mL, which is substantially outside the measurement range for far-UV CD, and there are limitations with respect to diluent choice, since formulation buffer cannot be used if it contains a significant level of chromophores. These data confirm that diluting the sample with PBS, an acceptable, well-characterized buffer for far-UV CD analysis, has no impact on the structure of the protein.

Analyzing Similarity across Both Dilution Series
In a final analysis, the two datasets were merged to look at similarity with respect to both concentration and buffer. Figure 8 shows the second derivative spectra for all of the samples; Table II shows the associated HOS data.

These data illustrate that the secondary structure of the mAb is extremely consistent, regardless of concentration or buffer. When compared with the 5 mg/mL sample in the formulation buffer, the similarity of the 1 mg/mL samples in formulation...
buffer are in the range of 98.2 to 98.5%, while those of all the other samples, in both the formulation and PBS buffer, are between 99.0 and 99.9%, indicating that the structure of the mAb is highly comparable across all the samples. The ability of MMS to measure across the full concentration range, without interference from either buffer, makes it extremely valuable for elucidating differences between data sets and for effective buffer screening. Here, the results indicate that any discrepancies observed between far-UV CD and MMS data are not attributable to either concentration or buffer components.

Conclusion
Instrumentation developed to characterize the secondary structure of proteins across a wide range of conditions, most especially over a broad concentration range, is particularly helpful to biopharmaceutical scientists. The development and manufacture of biopharmaceuticals calls for a detailed understanding and control of the drug entity, within the native formulation environment, and through manufacture and storage to the point of patient delivery. The use of different measurement techniques, especially under different protein conditions, significantly complicates the interpretation of and the analytical workflow. In particular, the comparison of datasets produced using alternative technologies can lead to discrepancies between results that cannot be attributed to the sample with any certainty.

The ability of MMS to measure with high reproducibility and repeatability, across a very wide concentration range, and to generate data unaffected by formulation excipients and additives, is extremely valuable within this context. This capability significantly reduces the need to compare and transfer specifications from one technique or instrument to another, thereby simplifying and nullifying risk in the analytical workflow, while at the same time reducing the cost associated with the purchase and ongoing maintenance of multiple instruments. Such flexibility makes MMS superior to alternatives such as far-UV CD and FT-IR for the essential task of characterizing the secondary structure of protein, from the earliest stages of drug delivery through to commercial biopharmaceutical manufacture and release testing.

References

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