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3D front face solid-phase fluorescence spectroscopy combined with Independent Components Analysis to characterize organic matter in model soils

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ABSTRACT

Soil organic matter (SOM) is a very complex and heterogeneous system which complicates its characterization. In fact, the methods classically used to characterize SOM are time- and solvent-consuming and insufficiently informative. The aim of this work is to study the potential of 3D solid-phase front face fluorescence (3D-SPFFF) spectroscopy to quickly provide a relevant and objective characterization of SOM as an alternative to the existing methods.

Different soil models were prepared to simulate natural soil composition and were analyzed by 3D front-face fluorescence spectroscopy without prior preparation. The spectra were then treated using Independent Components Analysis. In this way, different organic molecules such as cellulose, proteins and amino acids used in the soil models were identified.

The results of this study clearly indicate that 3D-SPFFF spectroscopy could be an easy, reliable and practical analytical method that could be an alternative to the classical methods in order to study SOM.

The use of solid samples revealed some interactions that may occur in natural soils (self-quenching in the case of cellulose) and gave more accurate fluorescence signals for different components of the analyzed soil models.

Independent Components Analysis (ICA) has demonstrated its power to extract the most informative signals and thus facilitate the interpretation of the complex 3D fluorescence data.

1. Introduction

Soil organic matter (SOM) has been directly and positively related to soil fertility and agricultural productivity potential. It is an important indicator of soil health as it plays a role in many key functions:

- it stores and supplies many nutrients needed for the growth of plants and soil organisms;
- it reduces the risk of erosion nutrient leaching;
- it prevents or minimizes soil compaction;
- it retains carbon from the atmosphere;
- it reduces the negative environmental effects of pesticides, heavy metals, and many other pollutants.

However, SOM has been called "the most complex and least understood component of soils" [1]. Magdoff considers SOM to be diverse organic materials, such as living organisms, slightly altered plant and animal organic residues, and well-decomposed plant and animal tissues that vary considerably in their stability and susceptibility to further degradation [2]. SOM is complex because it is heterogeneous (non-uniform) and, due to biological factors under which it was formed, it does not have a well-defined chemical and physical structure which makes its characterization difficult. In fact, classically, the characterization of SOM requires multiple extractions and fractionations [3–6] that use large volumes of solvents and long sample processing times, introducing the possibility for chemical alteration of the initial material. In addition, these methods of fractionating SOM into humic acid, fulvic acid, and humin are no longer considered meaningful because the obtained fractions are heterogeneous, non-reproducible and artificially defined (these fractions do not really exist as definite entities in soils) [7].

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Synchronous and classical three dimensional fluorescence spectroscopy have been widely used for dissolved organic matter [8,9], water extractable organic matter [5] and the characterization of humic and fulvic acids [4,10–12]. However, despite the interest and the efficiency of fluorescence spectroscopy in the study of dissolved organic matter, this technique has not been used to characterize the organic matter in solid samples.

Front-face fluorescence spectroscopy is a technique suitable for the analysis of concentrated, opaque or even solid samples. The advantages of this technique are its speed of analysis, lack of solvents and reagents, and requirement of only small amounts of sample. In addition, it is a non-invasive, highly selective and sensitive technique. The front-face (FF) technique has been shown to be able to provide accurate results in solid samples such as pharmaceutical products [13,14], semi-hard cheeses [15], crushed nuts and sesame seeds [16] and chicken meat [17].

The use of powder samples is a challenge [18,19]. Indeed, in addition to the signal saturation from the undiluted samples, the Rayleigh scattering in this case is much higher than the maximum intensity of the fluorescence signals which may mask the spectra of the sample components.

The interpretation of fluorescence spectral data is complex due to the presence of many fluorophores in the same excitation–emission-matrix (EEM). Different chemometric methods such as PARAFAC, MCR-LS and PLS [9,20–22] have been used in order to analyze the fluorescence data. In this paper, Independent Components Analysis (ICA), was applied to the 3D-front-face fluorescence spectra to facilitate their interpretation.

ICA aims at recovering the underlying source signals and their proportions from a set of mixed signals based on the assumption that these source signals are statistically independent [23]. The general ICA model is given by [24,25]

$$X = A \cdot S$$

where **X** is the matrix of observed spectra, **S** is the matrix of unknown "pure" source spectra and **A** is the mixing matrix of unknown coefficients, directly related to the corresponding proportions.

Based on the Central Limit Theorem, ICA assumes that statistically independent source signals have intensity distributions that are less Gaussian than their mixtures [24,25]. For this reason, ICA aims to maximize the non-gaussianity of the extracted signals.

The JADE (Joint Approximate Diagonalization of Eigenmatrices) [26] algorithm for ICA was used in this study. JADE maximizes the independence of the extracted signals by a joint diagonalization of matrices of the fourth-order cumulants calculated from the data. Fourth-order cumulants are measures of the non-gaussianity of combinations of the signals. The cumulants calculated from two pure independent signals are zero while those of mixed signals are non-zero. More details on the JADE algorithm can be found in [27].

The aim of this work was to study, using different mixtures of pure compounds (commercial powders) to simulate the composition of natural soils, the potential of 3D solid-phase front face fluorescence (3D-SPFFF) spectroscopy to quickly provide a relevant characterization of SOM as an alternative to the classical methods to avoid solvent use and sample preparation.

2. Materials and methods

2.1. Chemicals

All chemicals: Bovine serum albumin (BSA), lignin, sand (50–70 mesh), kaolinite (natural), charcoal (norit, type darco), cellulose, oil and grease in soil, L-tyrosine and L-phenylalanine were purchased from Sigma-Aldrich (St. Quentin Fallavier, France).

Table 1
Mixture design.

Experiments	BSA(%)	Oil and grease in soil (Lipids%)	Cellulose(%)
1	0	0	50
2	20	0	30
3	0	20	30
4	20	20	10
5	10	0	40
6	0	10	40
7	20	10	20
8	10	20	20
9	10	10	30
10	5	5	40
11	15	5	30
12	5	15	30
13	15	15	20

2.2. Experimental design

Different mixtures of pure compounds were prepared, in which the proportions of three pure compounds were varied: BSA and lipids (oil and grease in soil) between 0 and 20% and cellulose between 0 and 50% of the total organic matter (50%).

The mineral fraction (sand + kaolinite) was kept invariable at 40%. Similarly for lignin and charcoal that were maintained at 5% each.

The choice of the proportions of the pure compounds used in the model soils was based on the composition of natural soils. For example, the quantity of cellulose in natural soils is much higher than that of proteins (BSA) which does not exceed 20% of the total organic matter.

The Nemrod software [28] was used to generate a mixture design consisting of 13 different experiments as given in Table 1.

2.3. 3D-SPFFF analysis

Fluorescence landscapes (3D spectra) were measured directly on the samples without prior preparation, using a spectrofluorometer (LS45, Perkin-Elmer) equipped with a xenon lamp source, excitation and emission monochromators and a front-face sample-cell holder. Measurements were carried out using quartz cuvettes. The instrumental settings were excitation wavelengths 230–500 nm (step 4 nm) and emission wavelengths 250–560 nm (step 0.5 nm). Excitation and emission monochromator slit widths were set at 10 nm. Emission monochromator scan speed was 800 nm s^{-1}. A photomultiplier (PM) voltage of 650 V was used to avoid detector saturation. Unlike the other samples, BSA was analyzed with an 8% transmission attenuator (attenuator – approximately 8% Perkin-Elmer) on the excitation beam.

The data corresponding to each pure compound (BSA, cellulose, lignin, lipids, kaolinite, sand and charcoal) and each mixture (13 mixtures (see Table 1)) were arranged in a (68×618) matrix.

All the elementary matrices of the pure compounds and the mixtures were gathered together in a $(20 \times 68 \times 618)$ 3-way cubic array (20 spectra corresponding to the 13 mixtures and 7 pure compounds, 68 excitation wavelengths and 618 emission wavelengths). The cube of data was unfolded to give a (20×42024) matrix in order to apply ICA.

2.4. Spectra pretreatment

The two bands corresponding to the first- and the second-order Rayleigh scattering were detected and eliminated using the method proposed by Bahram et al. but without interpolation so as to avoid the addition of any information to the original spectra [29].

Table 2
Chemical interpretation of IC signals calculated by the JADE algorithm.

IC	Wavelength region	Possible fluorophores	References
1	λ_{ex}=230–250 nm, λ_{em}=300–420 nm λ_{ex}=250–300 nm, λ_{em}=330–490 nm λ_{ex}= 305–375 nm, λ_{em}=350–480 nm	Cellulose	[42–49]
2	λ_{ex}=250–310 nm, λ_{em}=310–380 nm	BSA	[5,33–37]
3	λ_{ex}=285–310 nm, λ_{em}=330–380 nm	Protein_like	
	λ_{ex}=310–450 nm, λ_{em}=375–530 nm	Free and protein-bound amino acids	
4		Rayleigh scattering	
5			

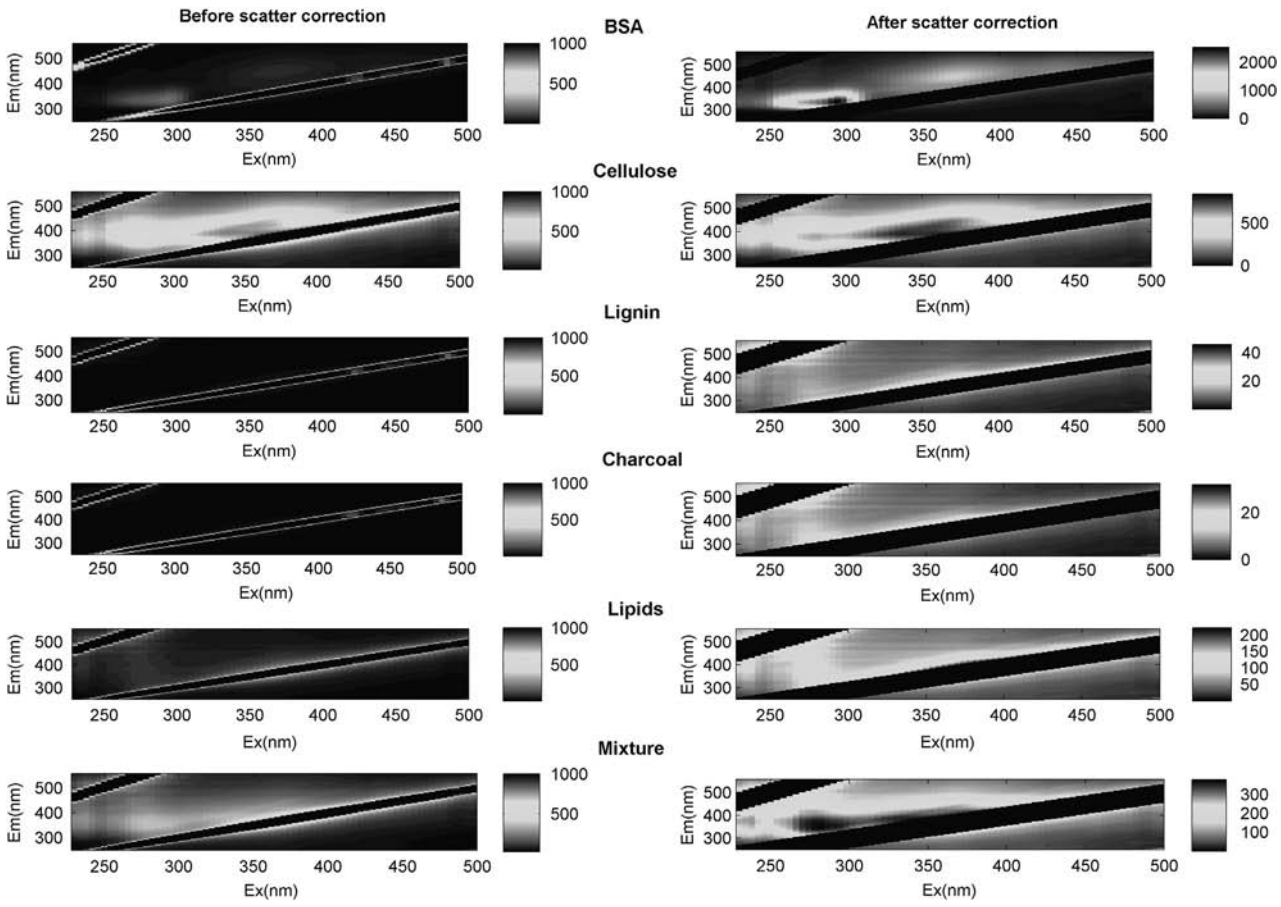

Fig. 1. Fluorescence excitation–emission matrices (EEM) obtained by 3D –SPFFF spectroscopy for commercial powders of BSA, cellulose, lignin, charcoal, lipids and a mixture example before (left) and after (right) Rayleigh correction.

3. Chemometrics methods

JADE was used to analyze the unfolded cube of EEMs (Table 2). The ICA_by_Blocks procedure uses JADE to determine the optimal numbers of independent components (ICs) to apply in the final ICA decomposition [30–32]. ICA_by_Blocks selects the optimal number of ICA signals to extract from a data matrix as being the number of signals found to be highly correlated in two representative subsets of the data matrix.

4. Results and discussion

4.1. Spectra

Fig. 1 shows the excitation–emission matrices (EEMs) of the different pure organic compounds and the EEM of an example of a soil model (Experiment 2 in Table 1) before and after scatter correction.

First- and second-order Rayleigh scatter signals do not contain any relevant chemical information. However, these bands can create problems when their intensity exceeds that of the informative fluorescence signals. In this case, the scatter complicates the analysis of the EEMs. As can be clearly seen in Fig. 1, the fluorophores are in some cases almost completely hidden (see BSA and mixture) by the Rayleigh scatter.

The BSA has a very intense peak at around λ_{ex}=250–310 nm, λ_{em}=310–390 nm [5,33–37] and another one less visible with a maximum at about λ_{ex}=370 nm, λ_{em}=450 nm.

In previous studies that have examined the fluorescence of proteins in solution [38–41], it has been shown that aromatic amino acids (tryptophan, tyrosine and phenylalanine) give a single peak in the region of the 'protein-like' fluorescence. However, in our case, BSA, in its commercial powder forms, presents two peaks.

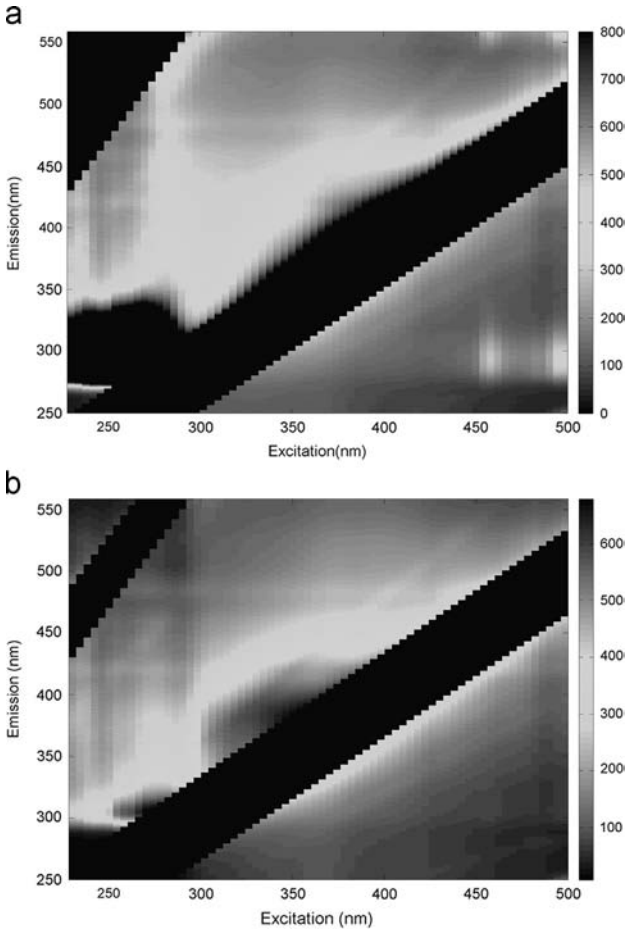

Fig. 2. Fluorescence excitation–emission matrices (EEM) obtained by 3D –SPFFF spectroscopy for commercial powders of phenylalanine (a) and tyrosine (b).

In Fig. 2a and b, two peaks can be seen in the EEM of tyrosine and phenylalanine commercial powders which is in accordance with the results found with BSA (see Fig. 6c where the scale was adjusted to make the second peak more visible). However, the same samples analyzed in aqueous solution give a single peak which confirms the existing studies (figures are not shown).

The EEM of cellulose shows a wide fluorescent region that can be divided into a peak around λ_{ex}=230–250 nm and λ_{em}=350–440 nm, a shoulder at λ_{ex}=250–300 nm, λ_{em}=330–480 nm and a peak about λ_{ex}=305–450 nm, λ_{em}=350–500 nm (see Fig. 1).

In the literature, depending on its source (wood, bagasse, cotton, etc.), cellulose has one to three characteristic peaks [42–49].

In the spectrum of lignin, we note that there is almost no fluorescence. However, in previous studies, lignin analyzed in solution fluoresces [50,51]. In our case, when analyzed in powder form, the fluorescence of lignin is greatly reduced due to the presence of strong light-absorbing chromophores [52].

There is no fluorescence for charcoal. In fact, as with lignin, this standard is a dark-colored solid that contains a high concentration of light-absorbing components which absorb excitation light and/or fluorescence emission [52].

In the mixture spectrum (Fig. 1), there is a large area of fluorescence that, at a first glance, contains the fluorophores of the pure compounds (BSA and cellulose). The interpretation of such spectra containing mixed signals is difficult. For this reason, ICA should facilitate the interpretation of the data by extracting the signals containing specific wavelength zones corresponding to individual pure compounds.

4.2. ICA analysis

The ICA_by_Blocks method was applied on the unfolded matrix with B=2 blocks and A_{max}=10. The correlation plot presented in Fig. 3 shows that after extracting 5 ICs, the curves of correlation coefficient drop. Thus, 5 ICs were used in the final ICA model.

Observing the spectra of the pure compounds (Fig. 1), we note that only two compounds fluoresce (there is not a lot of fluorescence for lipids). However, according to Fig. 3, there are five ICs to be extracted. In fact, not all IC signals correspond to chemical components; some ICs may result from variations in base line and residual Rayleigh diffusion (not shown here); and others may correspond to different physico-chemical phenomena associated with the same chemical compound [53].

4.3. Extracted independent components

IC1 (Fig. 4) is related to cellulose [42–49]. It can be seen that the pure compounds other than cellulose have the lowest proportions (Fig. 4a).

According to the distribution of the samples, the proportion values can be clearly seen to depend on the cellulose concentration in each mixture. The cellulose fluoresces more in mixtures (probably due to interactions with other constituents). This can be seen from the fact that cellulose at 100% is in fact situated between 20% and 10%. This may be due to self-quenching: the intensity of fluorescence is proportional to the concentration of the fluorophores over a certain concentration range. However, at high concentrations of fluorophores the proportionality is no longer satisfied, because of significant collisional quenching between the molecules of the fluorophore themselves [54].

IC2 (Fig. 5) is associated to BSA that produces fluorescence in the 'protein-like' region [5,33–37]. In fact, this pure compound has the highest proportion value (Fig. 5a).

The three mixtures containing higher BSA proportions (20%) have higher proportion values (Fig. 5a). However, the pure compounds (except BSA) have the lowest values. The quantities of BSA used in the mixtures (with a maximum of 20%) are smaller than those of cellulose (Fig. 4a) which may explain why the difference between the proportion values corresponding to the different mixtures is less clear.

The fluorescence intensity of BSA is not proportional to its concentration in each mixture, probably due to fluorescence quenching (bimolecular processes that reduce the fluorescence quantum yield without changing the fluorescence emission spectrum); that can result from transient excited-state interactions (collisional quenching) or from formation of non-fluorescent ground-state species [55].

The IC3 signal plot (Fig. 6b) shows two peaks. The first one (λ_{ex}=285–310 nm, λ_{em}=330–380 nm) is in the 'protein-like' region and is slightly shifted compared to the peak of BSA found in IC2. The second one, which is more visible, (λ_{ex}=310–450 nm, λ_{em}=375–530 nm) is at higher excitation/emission couple (see IC3 in Table 1).

These excitation/emission wavelength couples—generally considered as humic-like compounds [33]—were also reported to be the characteristic of complex products, such as melanoidins and lipofuscins, derived from protein, lipid and sugar oxidation [56–58].

The IC3 proportions plot (Fig. 6a) shows that pure compounds such as BSA and cellulose have the highest values corresponding to these fluorophores, while charcoal and lignin have the lowest values.

In Fig. 6, the excitation–emission spectrum of the BSA is shown in the same scale as that of cellulose in Fig. 4 so as to make the second peak visible which otherwise is almost invisible because the intensity of the peak is found in the 'protein-like' region.

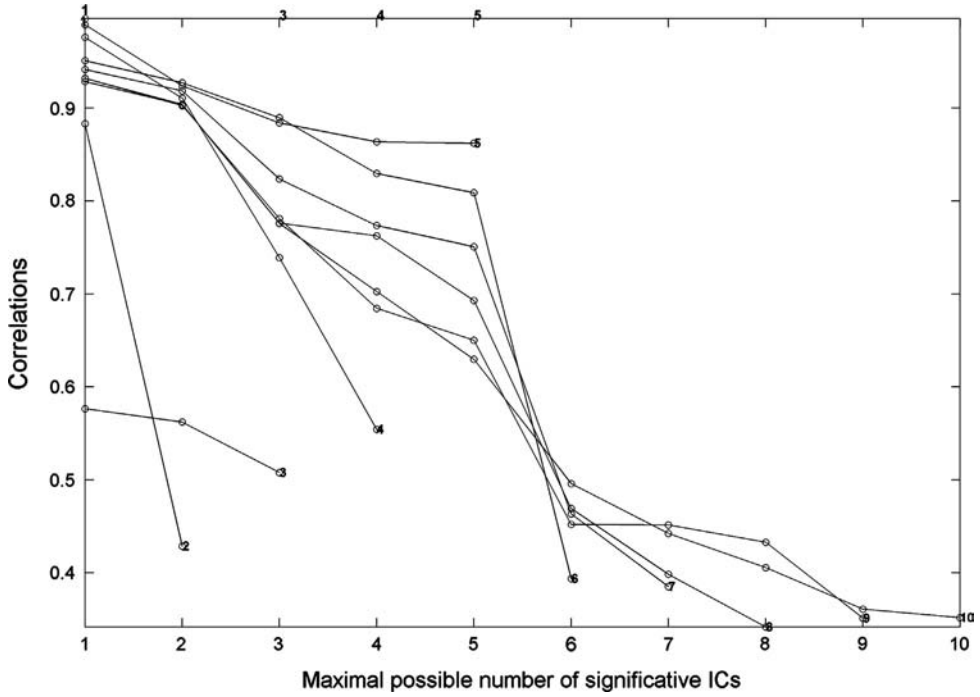

Fig. 3. Correlations between signals extracted from the two blocks.

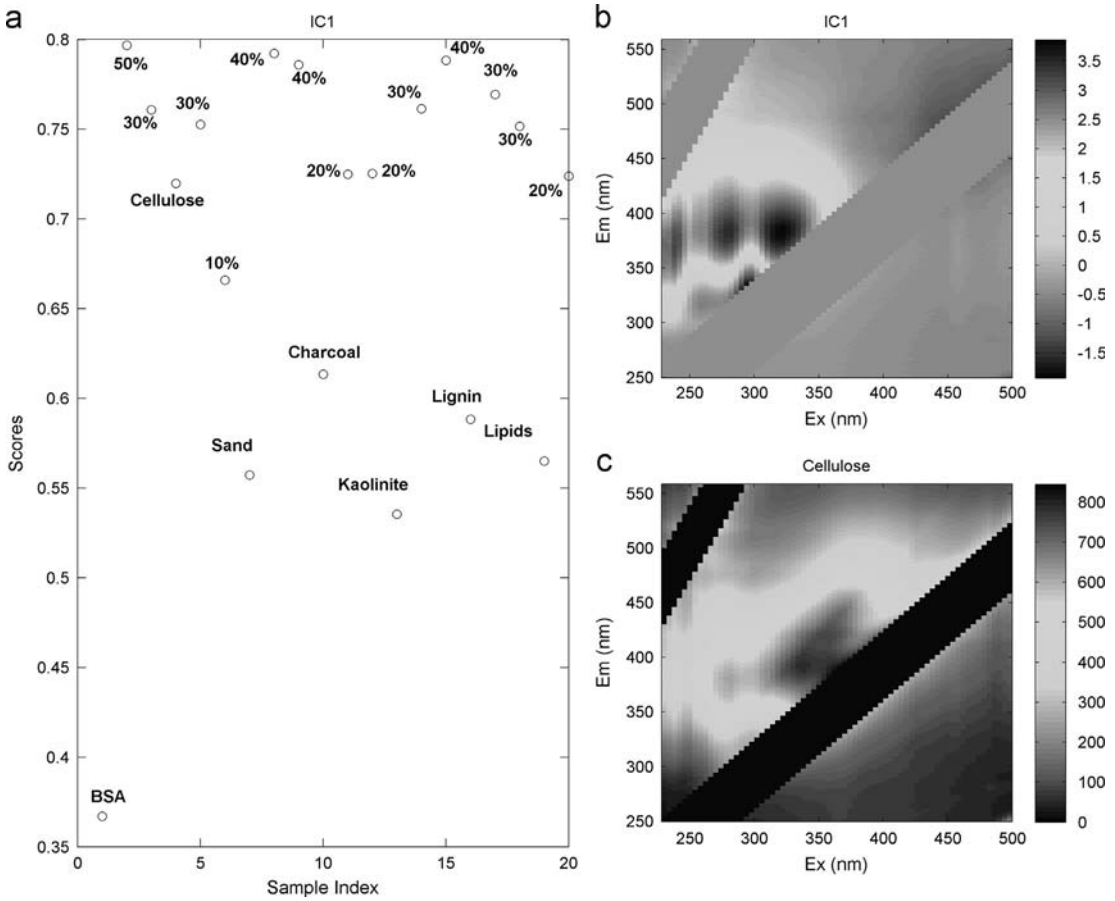

Fig. 4. Scores (a) and signal (b) of IC1 and EEM of cellulose (c).

This part of the signal is characteristic of the powder form of the BSA. In fact, as discussed in Section (4.1), BSA in aqueous solution shows a single peak in the protein-like region.

The second peak of the BSA spectrum, found at higher emission wavelengths, overlaps with that of cellulose and this is probably the reason why the proportion value of cellulose is also high (see

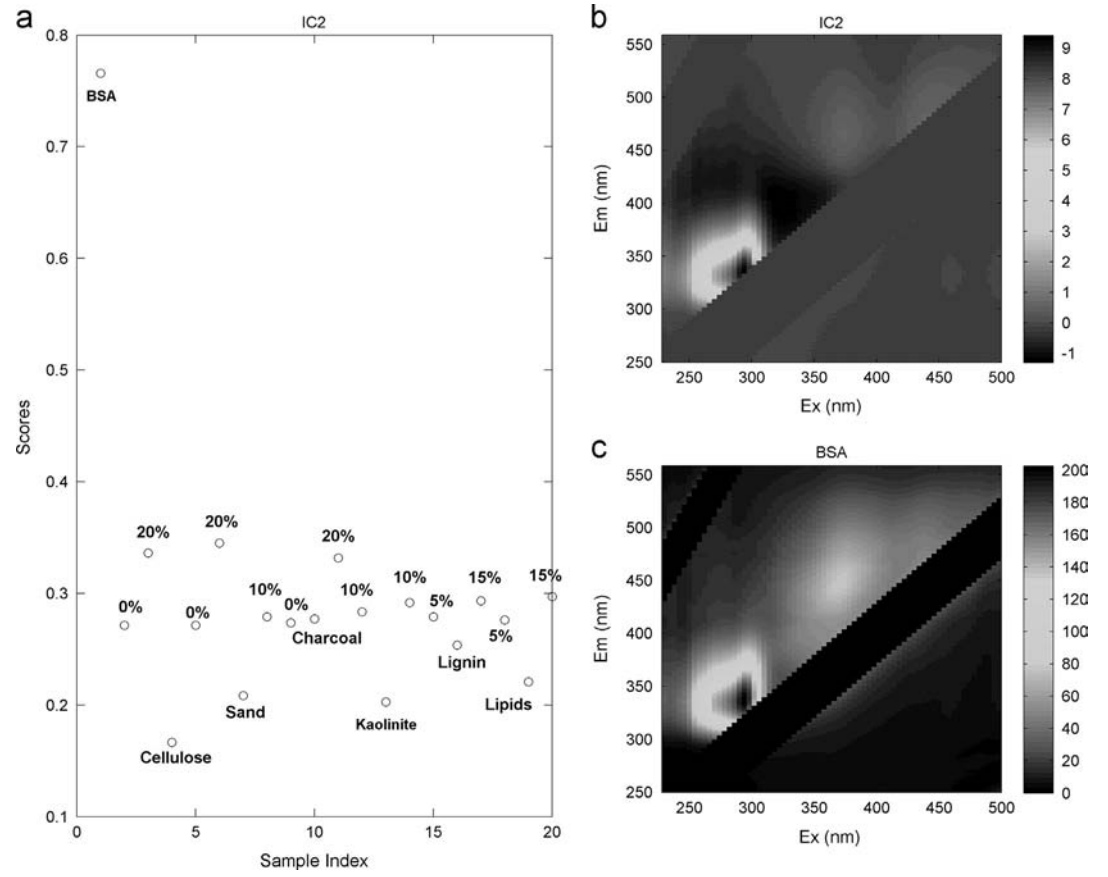

Fig. 5. Scores (a) and signal (b) of IC2 and EEM of BSA (c).

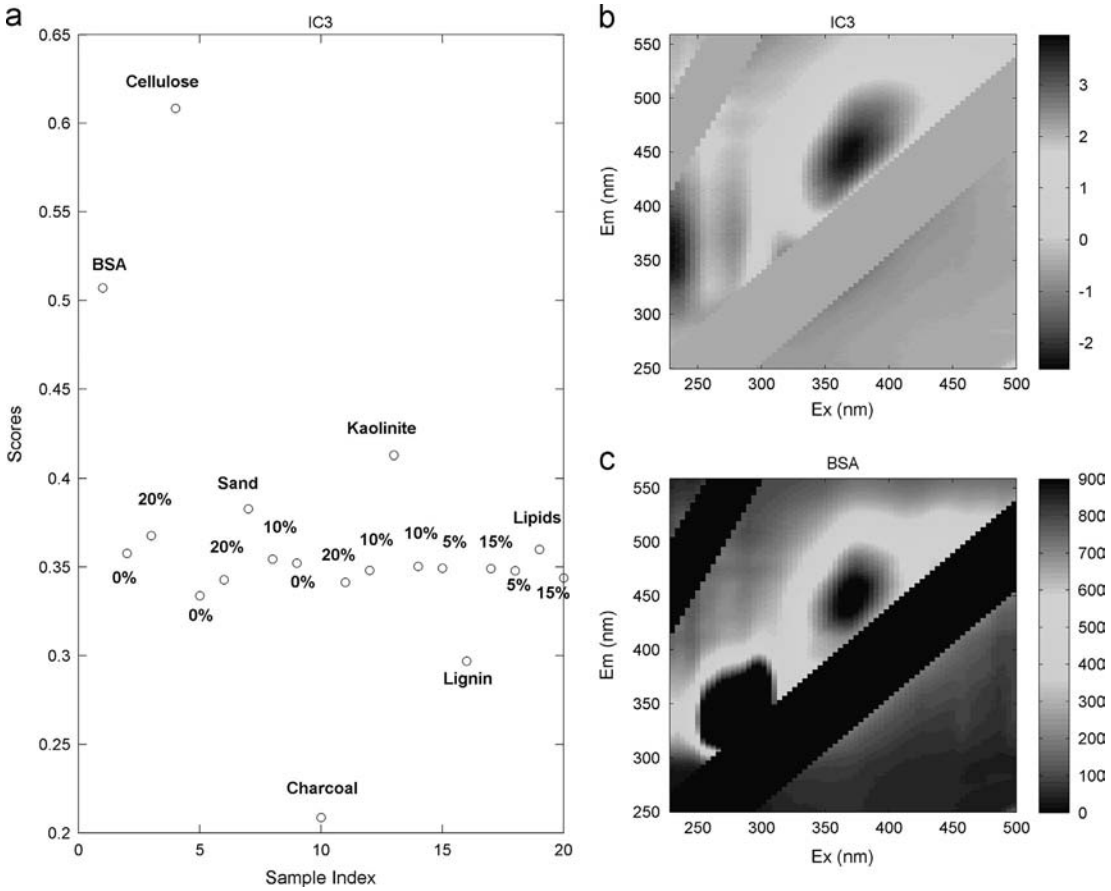

Fig. 6. Scores (a) and signal (b) of IC3 and amplified EEM of BSA (c).

Figs. 4c and 6c). Therefore, IC3 which corresponds to the second peak cannot be used to characterize BSA and cellulose.

Regarding the results obtained with the mixtures that simulate the composition of natural soils, 3D-SPFFF spectroscopy could be considered as a useful, simple and quick technique to characterize organic matter. In fact, using this technique coupled with Independent Components Analysis, several organic molecules such as cellulose, proteins and amino acids (major components of organic matter) can be identified. However, the quantification is difficult due to the problems of quenching and interaction between the compounds in the mixture.

In addition, when applied to solid samples, this technique gives results that better approximate the interactions that occur in natural soil, which is not the case for the samples analyzed in solution or after different extractions by solvent. In fact, it has been demonstrated in this study that the fluorescence of protein (BSA) and amino acids (tyrosine and phenylalanine) is different depending on the powder form or solution of the analyzed sample.

5. Conclusion

The present study aimed to evaluate the feasibility of 3D-fluorescence spectroscopy for the characterization of organic constituents of soils using simplified models. The observation of interactions between some compounds of the model soils shows that more knowledge is necessary about the phenomena of quenching and interactions between the compounds in the mixture before going on to analyze real soils samples.

3D front-face solid-phase fluorescence spectroscopy has been shown to be an easy, reliable and practical analytical method to study soil organic matter and especially lightly-colored matrices even if it still has some limitations for quantification due to interactions between the different constituents. Nevertheless, these problems do not affect the qualitative and, in some cases, semi-quantitative characterization of model soils.

The present study has also shown that ICA is a powerful tool for the decomposition of front-face 3D-fluorescence spectra and extraction of the constituent signals presenting specific wavelength zones corresponding to the individual fluorophores or to interpretable artifacts which facilitate the interpretation of the results.

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