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UV/VIS SPECTROSCOPY AS A METHOD TO CHARACTERIZE WELL WATER QUALITY

UV/VIS-spektroskopi som metod att karakterisera brunnsvattenkvalité

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Abstract

The applicability of UV/VIS spectroscopy for the characterization of well water quality was studied by analyzing 219 private wells from sparsely populated areas of Finland. In addition to conventional water quality indicators, such as conductivity, nitrate and DOC, a series of UV/VIS spectroscopic parameters were determined: A-224, A-254, A-280, A-365, A-436, the area under 250–350 nm, the area under 250–500 nm, SUVA-254, Molar Absorbance-280, Specific Color, A2/A3 and A2/A4, in order to find the most proper surrogate for the organic matter amount and the best indicator of organic matter quality. Three parameters, A-254, A-280 and the area under 250–350 nm were found to be applicable as surrogates for conventional aggregate organic matter indicators, giving rough approximations of organic content in well waters. A-224 can be used to estimate roughly the nitrate concentration in well water samples. A2/A4, SUVA-254 and Molar Absorbance-280 correlated positively with the high molecular weight fraction of NOM and therefore indicate surface water percolation or soil organic matter leaching into the wells. A2/A4 was found to be the most appropriate bulk spectroscopic parameter for the characterization of NOM structure and origin from wells with low and high dissolved organic matter content.

Key words – groundwater, UV/VIS, DOC, leaching, DOM, SUVA, HPLC-SEC

Sammanfattning

I denna artikel undersöktes möjligheterna att använda UV/VIS-spektroskopi för att karakterisera kvalitén av brunnsvatten genom analys av vatten från 219 privata brunnar i glest befolkade delar av Finland. Förutom vanliga indikatorer på vattenkvalité, såsom elektrisk konduktivitet, nitrat och DOC användes även parametrar från UV/VIS-spektroskopi; A-224, A-254, A-280, A-365, A-436, arean under 250–350 nm, arean under 250–500 nm, SUVA-254, Molar Absorbance-280, Specific Color, A2/A3och A2/A4 för att hitta det bästa substitutet för halten av organiskt material och den bästa indikatorn för kvalitén av organiskt material. Resultaten visade att A-254, A-280 och arean under 250–350 nm, kunde användas som substitut för konventionella indikatorer för organiskt material och A-224 kan användas för att erhålla en ungefärlig koncentration av nitrat. A2/A4, SUVA-254 och Molar Absorbance-280 var starkt korrelerade med fraktioner av NOM med hög molekylärvikt och är därför lämplig att användas som indikator på inläckage av ytvatten eller organiskt material från jordzonen. A2/A4 visade sig vara den mest lämpade bulkparametern för att karakterisera NOM struktur och ursprung från brunnar med både låg och hög halt av löst organiskt material.

1 Introduction

Well water samples contain higher or lower amounts of dissolved organic compounds and inorganic salts that are of natural or anthropogenic origin. The anthropogenic influence on a well is best seen in its increased nitrate concentration due to leaching from the soil caused by over fertilization or improper wastewater management (Korkka-Niemi, 2001; Szabo et al., 2009). The organic matter amount and quality of a well characterized by high performance liquid size exclusion chromatography (HPLC-SEC) can also be an indicator of the percolation of surface water or wastewater into a well and used for the identification of the wells exposed to contamination. (Szabo et al., 2009; Szabo and Tuhkanen, 2010).

The dissolved organic matter of a groundwater sample is mainly comprised of natural organic matter NOM, representing mostly the refractory humic and fulvic acids (Leenheer and Croué, 2003). There are three main sources of NOM in a natural water: 1. allochtonous sources, due to decaying and decomposing plant material; 2. autochthonous sources, which result from the internal production of a water body and mainly as a result of algal activities; and 3. anthropogenic sources, such as wastewater effluents. The amount of dissolved organic matter is given conventionally by water quality indicators, such as chemical oxygen demand COD and dissolved organic carbon DOC. These indicators, however, do not provide information on the organic matter quality.

UV/VIS spectroscopy is a technique based on the property of a molecule to absorb electromagnetic radiation in the UV/VIS range. This technique has been largely applied in water analysis, particularly for the quantitative estimation of aggregated organic parameters (DOC or COD), since NOM absorbs light over a wide range of wavelength and inorganic compounds do not absorb light significantly over 230 nm (Korshin et al., 1997; Uyguner and Bekbolet, 2005). Area under UV/VIS spectra between 250–350 nm and A254 (absorbance at 254 nm) were found to be applicable surrogates of COD and DOC, however, these studies used commercial humic acid solutions (Wang and Hsieh, 2001), surface waters and treated surface 55 waters (Vuorio et al., 1998; Liu et al., 2010) and wastewater effluents (Wu et al., 2006).

UV/VIS spectroscopy has also been used for the qualitative characterization of the NOM of water samples. Parameters, such as specific ultraviolet absorbance at 254 nm (SUVA-254), Molar Absorbance-280 and ratios A2/A3 (absorbance at 254 nm/ absorbance at 365 nm) and A2/A4 (absorbance at 254 nm /absorbance at 436 nm) are the most often used qualitative indicators (bulk parameters) of dissolved organic matter. SUVA-254 and Molar Absorbance -280 can be used for the estimation of aromaticity and the double bond densities of water samples, higher values presenting higher aromaticity and double bond densities (Chin et al. 1994, Peuravuori and Pihlaja, 1997, Frimmel and Abbt-Braun, 1999, Matilainen et al., 2011). It has also been shown that autochthonous NOM formed by the decomposition of algae, phytoplankton and bacteria has low Molar Absorbance-280 (150 L $(cm*mol)^{-1}$). On the other hand, the allochtonous NOM formed by the decomposition of higher plants has a considerably higher Molar Absorbance-280 (250–500 L $(cm*mol)^{-1}$); Molar Absorbance-280 can thus be an indicator of the contribution of terrestrial organic matter to the NOM of a water sample (Chin et al., 1994; Rosario-Ortiz et al., 2007). The molecular weight of NOM determined by HPLC-SEC correlates positively with Molar Absorbance-280 (Chin et al., 1994, Peuravuori and Pihlaja, 1997).

However, only a few of these studies concentrated on groundwater samples and in those studies the pre-treated and isolated groundwater NOM were studied, having probably changed properties when compared to the original whole-groundwater NOM (Frimmel and Abbt-Braun, 1999). In Finland wells present relatively high organic content with a mean that is approximately 3 mg L^{-1} DOC (Szabo et al., 2009). Nationwide well surveys have shown that high organic content under low pH condition is the most frequently found problem in Finnish wells (Korkka-Niemi, 2001). Therefore, UV/VIS could be used for the rapid screening of well water quality.

In this work we studied the applicability of UV/VIS as a fast instrumental method for the analysis of the quantity and quality of dissolved organic matter of whole well water samples without any pre-treatment. Additionally, we considered the assessment of nitrate concentration, since nitrate absorbs strongly at 220–230 nm (Thomas and Burgess, 2007) and can be present in high amounts in well water 85 samples. Our main aim was to find the UV/VIS spectroscopic parameters that are the best surrogates for aggregate organic matter and nitrate amounts, and the best indicators of organic matter quality in well water samples.

2 Materials and Methods

2.1 Wells, surface water and wastewater effluent samples

In total 219 private wells from sparsely populated areas of Finland were analyzed in this study. Each well was sampled once. The samples were collected in 1 l polyethene bottles and kept in a refrigerator until they were analyzed. The samples were analyzed within a maximum of five days of their sampling. For comparison, we analyzed two surface water samples and two wastewater effluent samples. Surface water 1 (lake Tuurujärvi) had relatively low DOC content (7 mg L^{-1}), while surface water 2 (Ahmausoja) had high DOC content (25 mg L^{-1}). One of the wastewater effluents was the final effluent of Tampere municipal wastewater treatment plant (a city of 220 000 inhabitants); the second wastewater effluent was an onsite septic system's effluent. The well waters, surface waters and wastewater effluents used in this study are described in detail elsewhere (Szabo and Tuhkanen, 2010).

2.2 Methods

Nitrate concentration was measured using the liquid chromatography of ions, with Dionex D 120 ion-chromatograph. The conductivity was measured by a WTW conductometer, which automatically corrects temperature to $+20^0$C. The dissolved organic carbon (DOC) of the samples was determined with a TOC analyzer SHIMADZU TOC-5000. For the determination of a relationship between qualitative spectroscopic indicators and the molecular weight of organic matter, we used the high performance liquid size exclusion chromatography (HPLC-SEC) data of the wells determined in a previous study (Szabo and Tuhkanen, 2010). HPLC-SEC separates the organic matter into fractions according to their molecular weight. For the well waters of this study we obtained seven fractions and grouped them as high molecular weight fractions, HMW (fractions I, II and III); intermediate molecular weight fractions, IMW (fractions IV and V) and low molecular weight fractions, LMW (fraction VI). The fractions are characterized quantitatively by the height of the peak maximum expressed in milli ampere units (mAU). SUM-254 in mAU represents the sum of the heights of all five peaks eluted and is a surrogate of DOC. The principle of the method and the well water chromatographs are described and presented in detail in Szabo and Tuhkanen, 2010. The absorbance values of the samples between 220–500 nm, including the values at 224 nm (A-224), 254 nm (A-254), 280 nm (A-280), 365 nm (A-365) and 436 nm (A-436) were determined with a Shimadzu UV 1601 spectrophotometer, using Eppendorf plastic cuvettes for the measurements in the range of 220 nm–1600 nm. From the absorbance and DOC values the qualitative bulk-parameters, such as SUVA-254 (A-254* $(DOC)^{-1}$), molar Absorbance at 280 nm (A-280*12*$(DOC)^{-1}$), Specific Color (A-436*$(DOC)^{-1}$), A2/E3 (A-254/A-365) and A2/A4 (A-254/A-436) were determined in order to obtain information on the NOM structure. In order to exclude the outliers (too high spectroscopic values for too low DOC, A-365 and A-436), DOC values smaller than 0.1 mg L^{-1}, respectively A-365 and A-436 values lower than 0.001 cm^{-1} were considered 0 and excluded from the spectroscopic data set. After exclusion, the data of 172 wells were analyzed.

2.3 Statistical analyses

For the statistical analysis of the data SPSS 15.0 (SPSS Inc.) was used. To assess the strength of the association between the parameters, Spearman's rank correlation was applied to the data set. Two levels of statistical significance were considered ($p < 0.05$ and $p < 0.01$). For the estimation of the relationship between the nitrate concentration and A-224, and the dissolved organic matter amount (DOC and SUM-254) and the surrogate spectroscopic parameters (A-254, A-280, the area under 250–350 nm and the area under 250–500 nm) linear and polynomial (quadratic and cubic) regression analyses were applied to the data set.

3 Results and Discussions

3.1 Quantitative DOM and nitrate surrogates

The statistical values of well water parameters analyzed in this study are presented in Table 1. The absorption spectra of four representative well samples are given in Figure 1. The well water UV/VIS spectra are broad, with no specific maxima, except the samples maximum in the spectra at the wavelength between 220–225 nm.

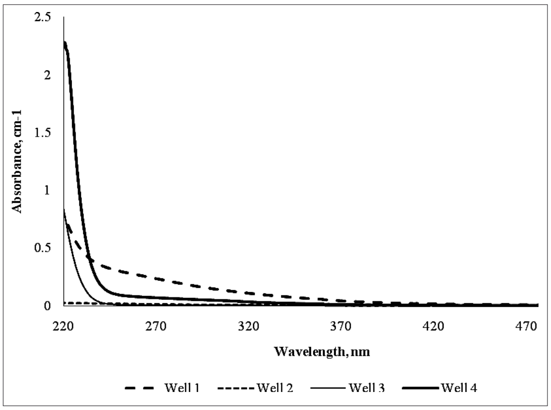

Figure 1. *The typical UV/VIS absorption spectra of wells with various DOC and nitrate content (Well 1: DOC=12 mg* L^{-1}*;* NO_3^-*=0 mg* L^{-1}*; Well 2: DOC=1.3 mg* L^{-1}*;* NO_3^-*=0 mg* L^{-1}*; Well 3: DOC=1.25 mg* L^{-1}*;* NO_3^-*=77 mg* L^{-1}*; Well 4: DOC= 9 mg* L^{-1}*;* NO_3^-*= 100 mg* L^{-1}*).*

Table 1. *Descriptive statistics of the well water parameters analyzed in this study.*

Parameter (unit)	N	Minimum	Maximum	Mean
Conductivity (µS cm^{-1})	169	40.00	3030.00	263.75
NO_3^- (mg L^{-1})	172	0.00	219.94	14.75
DOC (mg L^{-1})	172	0.33	11.78	2.70
SUM-254 (mAU)	172	0.00	29.90	5.41
HMW (mAU)	172	0.00	19.86	2.21
IMW (mAU)	172	0.00	12.70	2.44
LMW (mAU)	172	0.00	3.57	0.76
A224 (cm^{-1})	172	0.00	2.51	0.38
A254 (cm^{-1})	172	0.01	0.29	0.04
A280 (cm^{-1})	172	0.00	0.20	0.03
A365 (cm^{-1})	172	0.00	0.06	0.01
A436 (cm^{-1})	172	0.00	0.08	0.006
Area under 250–350 nm (arbitrary units)	172	0.71	32.19	5.75
Area under 250–500 nm (arbitrary units)	172	1.51	54.72	9.61
SUVA-254 ($L(g*cm)^{-1}$)	172	4.01	44.95	18.43
Molar Absorbance-280 ($L\ (cm*mol)^{-1}$)	172	39.49	393.04	167.85
SpecificColor-436 ($L\ (g*cm)^{-1}$)	171	0.00	19.13	3.10
A2/A3	172	1.04	12.57	4.79
A2/A4	168	1.10	59.60	11.53

According to Korshin et al. (1997), the inorganic chemicals present in natural waters absorb significantly at wavelengths up to 230 nm. In this study, the conductivity that represents the total dissolved solids in the samples did not correlate significantly with absorbance at 224 nm ($p > 0.1$). The nitrate concentration, however, did show significant correlation with A-224 nm (Spearman correlation coefficient 0.8, $p < 0.001$), indicating that A-224 could be used as surrogate parameter for nitrate. Linear and second order polynomial regression between nitrate concentration and A-224 nm gives R^2 of 0.58 and 0.59 only (Figure 2d), which indicates that A224 can only give a rough approximation of the nitrate amount in a well water sample.

As expected, DOC and SPH-254 correlate significantly with all quantitative spectroscopic parameters, with the single exception of A-436 (Table 2). Strong correlations between A-254 and the area under 250–350 nm and the DOC of surface waters and commercial humic acid solutions were previously found in a number of studies (Vuorio et al., 1998; Wang and Hsieh, 2001; Wu et al., 2006; Liu et al., 2010). In this study, however, only three spectroscopic parameters have strong correlation coefficients with aggregate organic matter indicators: A-254, A2-80 and the area under 250–350 nm. The strongest correlation is given by A-254, in contrast with previous results on dissolved commercial humic acids in which the area under the spectra between 250–350 nm was found to be the best surrogate for NOM (Wang and Hsieh, 2001). The relatively low R^2 values of linear and polynomial regression (0.64–0.69) between DOC and A-254 (Figure 2a), A-280 (Figure 2b) and the area under 250–350 nm (Figure 1c) show that these surrogate spectroscopic parameters give only approximations of DOC in well water samples. The regression analysis between SUM-254 and the quantitative spectroscopic parameters yielded slightly smaller R^2 values than those of DOC (not presented).

It should be also noted that there is a significant correlation between organic indicators and A-224, which shows the mutual interference of nitrate and dissolved organic matter on absorption at this wavelength.

Table 2. *Spearman's rank correlation coefficients between quantitative dissolved organic indicators and surrogate spectroscopic parameters.*

	A-224 (cm^{-1})	A-254 (cm^{-1})	A-280 (cm^{-1})	A-365 (cm^{-1})	A-436 (cm^{-1})	Area under 250–350 nm (arbitrary units)	Area under 250–500 nm (arbitrary units)
DOC (mg L^{-1})	0.442^*	0.784^*	0.771^*	0.554^*	0.068	0.741^*	0.518^*
SPH-254 (mAU)	0.502^*	0.828^*	0.808^*	0.607^*	0.135	0.786^*	0.603^*

*Correlation is significant at the 0.01 level.

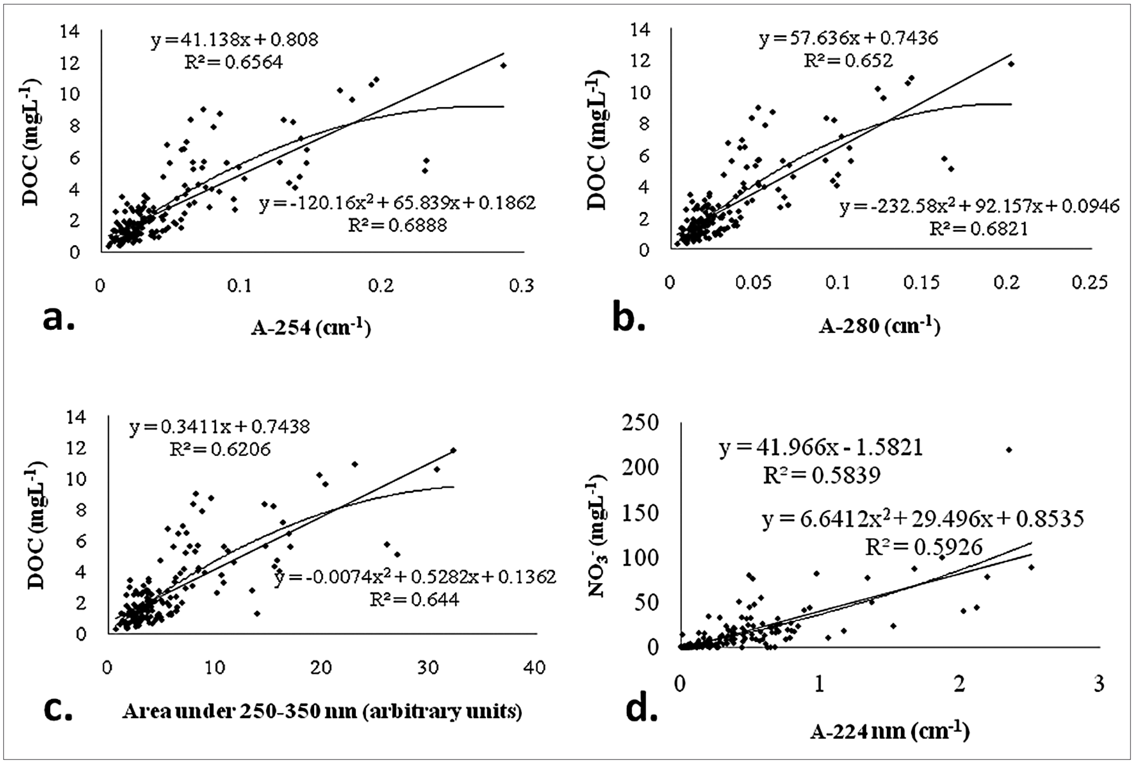

Figure 2. *Linear and second order polynomial regression between DOC and surrogate spectroscopic parameters (a, b, c) and between nitrate and A-224 nm (d). A higher order polynomial regression does not significantly change R^2.*

3.2 Qualitative (bulk) DOM parameters

The statistical values of the bulk spectroscopic DOM parameters of well waters are presented in Table 1. The same spectroscopic parameters of wastewater effluents and surface waters are presented in Table 3.

SUVA-254 and Molar Absorbance-280 show the lowest values for unprocessed onsite wastewater effluent and show its low aromatic content. Properly processed municipal wastewater effluent has three times higher SUVA-254 and Molar Absorbance-280 values than the onsite effluent suggesting that aromaticity concentrates during wastewater processing. Surface water samples showed the highest SUVA-254 and Molar Absorbance-280 values, which corresponded to the highest aromaticity and

Table 3. *The bulk spectroscopic parameters of wastewater effluents and surface waters analyzed in this study.*

	SUVA-254 L (g*cm)$^{-1}$	Molar Absorbance-280 L (mol*cm)$^{-1}$	Specific color L (g*cm)$^{-1}$	A2/A3	A2/A4
Municipal secondary wastewater effluent DOC = 9.25 mgL^{-1}	23.69	214.70	1.6	5.58	14.80
On-site wastewater effluent DOC = 88 mgL^{-1}	7.719	70.96	0.65	5.72	11.86
Surface water 1 DOC = 7 mgL^{-1}	43.299	370.59	4.66	4.49	9.30
Surface water 2 DOC = 25 mgL^{-1}	52.84	488.55	3.68	4.37	14.34

the highest allochtonous influence. Similar SUVA-254 and Molar Absorbance-280 values for different water types were obtained in previous studies (Chin et al., 1994, Peuravuori and Pihlaja, 1997, Frimmel and Abbt-Braun, 1999).

The mean SUVA-254 and Molar Absorbance-280 values of the wells are relatively low showing a mainly autochthonous character, and fall between the values of onsite and municipal wastewater effluents (Table 1); the same range of SUVA-254 and Molar Absorbance-280 for ground waters was obtained by Frimmel and Abbt-Braun (1999) and Chin et al. (1994). The maximum SUVA-254 and Molar Absorbance-280 values, however, are close to the surface water samples and present a clear allochthonous character.

For low DOC concentrations (under 2 mg L^{-1}, according to Wang and Hsieh, 2001) the UV/VIS absorbance of natural water samples may be low, in these cases relatively high random errors in both DOC and absorbance determinations might occur. These random errors can cause large deviations in the calculation of the qualitative spectroscopic parameters. The random errors are less significant for samples with high DOC and correspondingly higher UV/VIS absorbance. In order to get a clearer view on the spectroscopic properties of well waters, we separated the wells according to their DOC concentrations into two groups, respectively wells with low-DOC having DOC < 2 mg L^{-1} (n = 95) and high-DOC wells with DOC > 2 mg L^{-1} (n = 77). The Spearman correlation coefficients between the spectroscopic values and the other water quality indicators are given in Tables 4a and 4b.

The correlations of SUVA-254, Molar Absorbance-280 and Specific Color with DOC are significant and negative for the low-DOC group, which is expected due to the method for calculating these parameters. However, for the high-DOC group the correlation coefficients between DOC and SUVA-254 as well as Molar Absorbance-280 are insignificant, which suggests that at higher DOC values SUVA-254 and Molar Absorbance-280 do not depend on DOC. Specific Color keeps its negative correlation with DOC in the high-DOC group because yellowish-brownish compounds are found in relatively small amounts, even in wells with higher DOC. The significant positive correlations among SUVA-254, Molar Absorbance-280 and HMW, IMW for the high-DOC group are in accordance with previous studies showing that higher molecular weight NOM present higher SUVA-254 and Molar Absorbance-280 and correspondingly higher aromaticity (Chin et al., 1994; Peuravuori and Pihlaja, 1997; Uyguner and Bekbolet, 2005; Matilainen et al., 2011).

The spectroscopic ratios A2/A3 and A2/A4 are commonly used for the characterization of aquatic NOM. A2/A4 indicates the ratio between the absorption intensity of the UV absorbing functional groups and that of the VIS-absorbing yellow-brownish groups. For the surface water samples, it has been found that A2/A3 increases with a decrease in aromaticity (Peuravuori and Pihlaja, 1997). Our results on surface waters and wastewater effluents are in accordance with the results of previous studies, the less aromatic onsite wastewater effluent having the highest and the most aromatic surface water 1 having the lowest A2/A3 value (Table 3.). How-

Table 4 a. *Spearman's rank correlation coefficients for DOC < 2 mg L^{-1} (n=95 wells).*

	DOC	SUM-254	HMW	IMW	LMW
SUVA	0.440^{**}	0.109	0.204^{*}	0.143	–0.092
Molar Absorbance-280	0.479^{**}	0.039	0.141	0.073	–0.119
A2/A3	0.198	0.286^{**}	0.184	0.260^{*}	0.133
SpecColor	0.513^{**}	–0.202	–0.107	–0.179	–0.145
A2/A4	0.296^{**}	0.355^{**}	0.324^{**}	0.323^{**}	0.156

Table 4 b. *Spearman's rank correlation coefficients for DOC > 2 mg L^{-1} (n=77 wells).*

	DOC	SUM-254	HMW	IMW	LMW
SUVA	0.001	0.289	0.308^{**}	0.234^{*}	–0.015
Molar Absorbance-280	–0.014	0.270	0.288^{*}	0.212^{*}	–0.014
A2/A3	0.289^{*}	0.188	0.208	0.248	–0.010
SpecColor	0.423^{**}	–0.157	–0.199	–0.184	–0.031
A2/A4	0.461^{*}	0.393^{**}	0.448^{**}	0.398^{**}	0.029

**Correlation is significant at the 0.01 level; *Correlation is significant at the 0.05 level.

ever, with respect to the well waters of this study, we found no significant correlations ($p > 0.2$) between the indicators of aromaticity (SUVA-254 and Molar Absorbance-280) and A2/A3.

According to previous studies, groundwater samples show higher A2/A4 values than high NOM containing surface waters (Frimmel and Abbt-Braun, 1999, Uyguner and Bekbolet, 2005). We found the mean of the A2/A4 of the well waters studied is close to that of onsite wastewater effluent, but there were wells that also presented significantly higher or lower values than the surface waters (Table 1, Table 3). The general tendency for the well waters is that as the NOM amount increases, the A2/A4 increases, which is shown by the significant positive correlations between the aggregate organic matter indicators (DOC and SUM-254) and A2/A4 for both group of wells (Table 4a and 4b). Additionally, the A2/A4 ratio is significantly positively correlated to the HMW and IMW fractions, showing the tendency of A2/A4 to increase with the increase of molecular weight and aromaticity. This result contradicts the result of a previous study on commercial humic acids and surface waters which found that A2/A4 decreases with the increase of molecular weight (Uyguner and Bekbolet, 2005).

SUVA-254, Molar Absorbance-280 and A2/A4 tend to increase with the increase of molecular weight and correspondingly with the aromaticity of NOM from well water samples. These are thus potential bulk-parameters for the routine characterization of well water samples containing relatively high amounts or dissolved organic matter. The most suitable spectroscopic parameter is A2/A4 as it can be used for the qualitative characterization of the NOM of wells that have low as well as high dissolved organic matter content.

4 Conclusions

Spectroscopic indicators such as A-254, A-280 and the 254 area under 250–350 nm can be used as surrogates of aggregate organic matter indicators, such as DOC, but give only approximations of its values in wells. A-224 can be used for the rough estimation of nitrate concentrations in well water samples and, as such, indicates anthropogenic influence on the well. For wells containing a high amount of dissolved organic matter, SUVA-254 and Molar Absorbance-280 tend to increase with the increase of the molecular weight of NOM. Correspondingly, high SUVA-254, Molar Absorbance-280 and A2/A4 values indicate an allochtonous influence on the wells (soil/surface water percolation). A2/A4 was found to be the best bulk spectroscopic parameter for the characterization of NOM from wells that have low as well as high dissolved organic matter content.

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