Variation in the acidity of dissolved organic matter across Sweden during 35 years

(*Variationen i surheten av löst organiskt kol över Sverige under 35 år*)

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Variationen i surheten av löst organiskt kol över Sverige under 35 år – Slutrapport

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Summary

While acid deposition has declined, there are still many relatively acid streams and lakes in Sweden. Dissolved organic matter (DOM) influences the acidity of surface waters. This needs to be considered when targeting efforts to mitigate anthropogenic surface water acidification through liming.

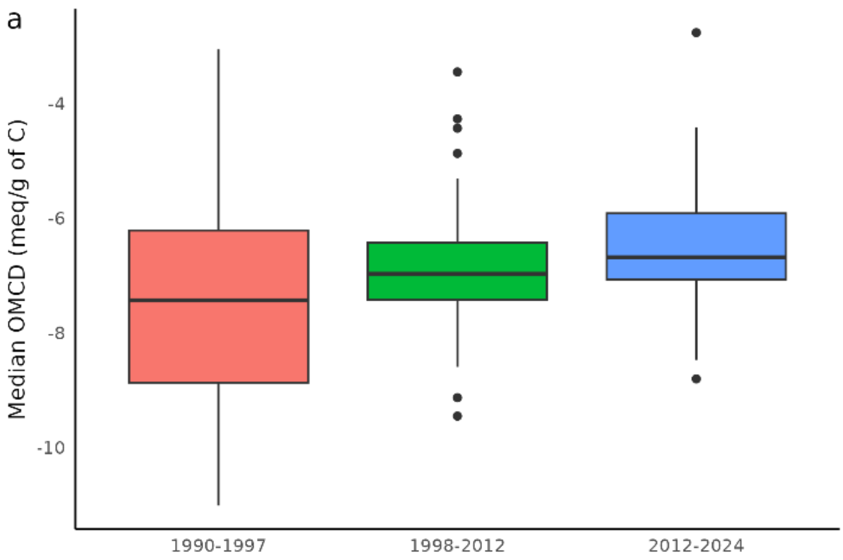
Reliably quantifying the degree of natural acidity related to DOM is a major scientific challenge. This involves determining both the natural concentration of DOM, and calculating how DOM influences pH. Fortunately, the national monitoring data on surface water chemistry also contains the information needed to quantify one aspect of the acidity of DOM, the organic matter charge density at pH 5.6 ($OMCD_{5.6}$). These data, over 42,000 samples analyzed between 1990 and 2024 from 136 national monitoring, were used to make that quantification in this project.

The variation in TOC, sVISa, organic charge and $OMCD_{5.6}$ and total organic charge within these stations and between stations was quantified. $OMCD_{5.6}$ stands out by having less variation between station medians. The median station $OMCD_{5.6}$ has changed over time in two thirds of the stations, with the direction of change more often positive than negative. The “increase” in $OMCD_{5.6}$ means that the organic matter became less charged, since organic matter is negatively charged. This decline in charge density would make a given amount of TOC lower the water pH less. A lower charge density in more recent years would also suggest a lower solubility of soil organic matter, since it is the negative charge of OM that contributes to its solubility.

While it is interesting to find variation in one feature of the acid base properties of DOM, this begs the question of how important this variation is for the pH of surface waters. The answer to this question varies from site to site. To provide an initial estimate of the significance of the observed variation in $OMCD_{5.6}$, we compared the amount of variation in negative charge (equivalents) that organic matter provides due to variation in $OMCD_{5.6}$, relative to the total amount of charge that organic matter provides. As a first approximation, the variation of the dissociation

properties changes the contribution of organic acids to the charge balance by +/-30%.

It will take more detailed analyses to reveal what this means for pH in specific cases. At this point, we can say that this degree of variation in the organic acid dissociation between water samples at the same pH is not consistent with the assumption that spatial and temporal variation in the organic acid dissociation properties of DOM do not need to be accounted for when assessing the role of organic acids in surface water chemistry.



Nyckelord: nyckelord 1, nyckelord 2

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1. Introduction

While acid deposition has declined, there are still many relatively acid streams and lakes in Sweden. Dissolved organic matter (DOM) influences the acidity of surface waters. This needs considered when targeting efforts to mitigate anthropogenic surface water acidification through liming.

Reliably quantifying the degree of natural acidity related to DOM is a major scientific challenge. This involves determining both the natural concentration of DOM, and calculating how DOM influences pH. Much of that challenge has focused on the quantity of DOM that would be present in streams and lakes without human influence from acid deposition and other human activities such as forest management and drainage. Changing DOM concentrations across Sweden in recent decades has received attention concerning assessing plans for liming to counteract surface water acidification. (e.g. Erlandsson et al, 2011), since both reduced acid deposition, forest management and drainage can increase DOM (Kritzberg et al., 2019). This will change the contribution of organic acids to the acidity of surface water.

Less attention, with the exception of a recent Norwegian study by Vogt et al. (2024), has been paid to whether the acid-base properties of DOM have been changing. This involves quantifying the degree to which a milligram of DOM influences pH. The lack of interest in this question can be attributable to the early success by SLU researchers in quantifying the acid base properties of Swedish surface water DOM over two decades ago. They demonstrated that a single set of parameters in a relatively simple equation described the organic acid properties of DOM quite well (Köhler et al., 2000). This worked for sites where it was tested across Sweden and Europe (Hruska et al., 2003). Thus, it was assumed in much subsequent Swedish surface water assessment work that differences in the acid-base character of organic carbon with respect to location, flow conditions and season did not need to be considered when defining the contribution of DOM to the acidity of surface water.

Since that original work was done, however, there have been shifts in a feature of DOM character that can be identified in national monitoring data- the color, which is routinely measured as absorbance at 420nm. This absorbance, when normalized to the concentration of DOM, is specific visible absorbance (sVISa). There was a widespread increase in specific absorbance (sVISa) in the decade between 1992 and 2002, followed by some decreases during the subsequent decade (Eklöf et al, 2021). More precisely, of just over 300 Swedish watercourses that are regularly monitored by national programs, DOM has been increasing continually in about 20% of them between 1990 and 2024 (Fig. 1). During three periods of 3-5 years, centered on 1997, 2007 and 2021, the proportion of streams with increasing DOM was higher. Nevertheless, at the same time as DOM was increasing in between 20 and 40% of the monitored streams in the 1990s, the color increased even more during the 1990s. Then color did not increase during the 2000s in many of the streams where DOM increased. As a result sVISa increased during the 1990s and then decreased in the 2000s (Fig. 1). These increases were scattered across the country from north to south. (Appendix Fig. A1 & A2). These changes in one aspect of organic carbon character, sVISAa, raises the question of whether another feature of organic carbon character, the acid-base dissociation properties has also been changing.

If that change in character is associated with a change in the acidity of DOM itself, this is of critical importance to the assessment of surface water acidification, and thus the SLU FOMA Acidification program. Fortunately, the national monitoring data on surface water chemistry also contains the information needed to quantify one aspect of the acidity of DOM, the organic matter charge density at pH 5.6 ($OMCD_{5.6}$). These data, over 42,000 samples analyzed between 1990 and 2024 from 136 national monitoring were used to make that quantification in this project.

This quantification indicates if and how the acid base properties of organic matter have changed over the course of 35 years when the amount and character of organic carbon (as indicated by DOM and sVISa) has changed at many stations. The fact that all the chemical analyses were made by the same SWEDAC accredited laboratory improves the chance that any changes found are not an artefact of the analytical methodology. The overall goal of this study is to determine whether changes in acid base properties of organic matter need to be considered when assessing the contribution of DOM to the acidity of water.

This project uses OMCD$_{5.6}$ to describe the organic acidity of DOM because it is the endpoint of alkalinity/acidity titration performed on surface water samples in many national surface water monitoring programs. It is at this titration endpoint of pH 5.6 where the charge balance between anions and cations is most precisely determined. While it would be more helpful to know the degree of dissociation across the range of natural pH in surface waters, that information is not collected routinely due to the costs involved. A key step in determining OMCD$_{5.6}$ from the available laboratory data archived and published on the MVM database (miljodata.slu.se/MVM/) is determination of all the weak acids and bases besides organic matter that are contributing to the charge balance. This information was determined using the Visual Minteq model (Gustafsson, 2023). As a further step in the project, relationships between eventual variations in OMCD5.6 were explored in relation to a range of catchment properties, as well as change over time.

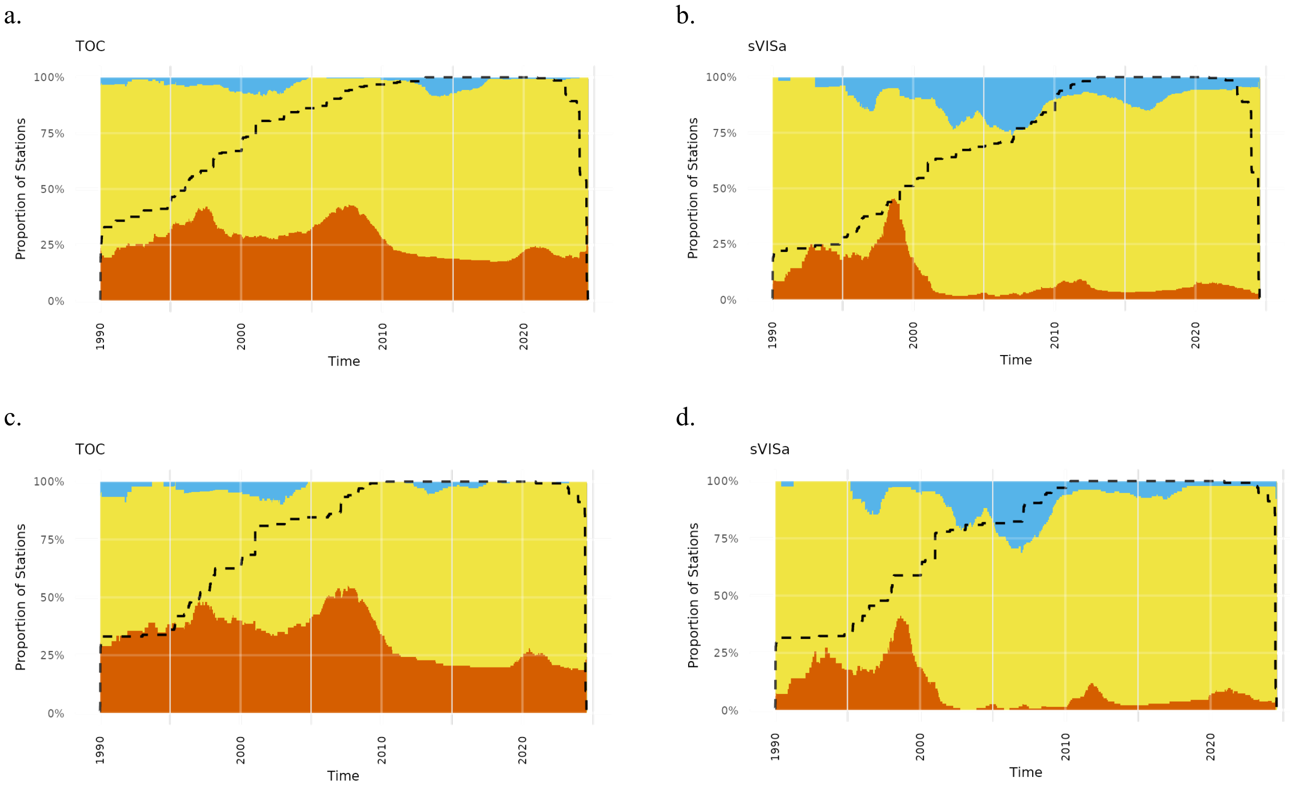


Figure 1. Proportion plot of TOC (a-b) and sVISa (c-d) trends across Sweden. This figure is adapted from Eklöf et al, 2020 by including several more years of data. Orange indicates an increasing trend, yellow is no trend and blue is a decreasing trend. On the left (b,d) all 316 stations that fit the initial selection criteria for this project of 10+ years of TOC data and < 5 percent urban area (see Methods for more details on these criteria).On the right (a,c) the 136 stations where charge density was modelled of this project (see Results for an explanation of which stations were modeled successfully).

2. Methods

This project use the detailed, high quality surface water chemistry data from national monitoring of lakes and watercourses to quantify the acid-base dissociation properties of dissolved organic matter across Sweden over the period 1988-2023. These environmental monitoring data have all been analyzed using SWEDAC accredited methods by the same laboratory, the SLU Department of Aquatic Sciences and Assessment Geochemistry Laboratory (slu.se/institutioner/vatten-miljo/laboratorier/vattenkemiska-laboratoriet/). The consistency of the analysis methods provides a unique opportunity to investigate these properties. Of particular interest for this study is the endpoint titration of alkalinity/acidity since it provides precise information on the pH and pCO2 of the water sample at the same time. The titration begins by bubbling the water sample with nitrogen gas to remove all dissolved CO2 from the sample. The pH is then titrated to pH 5.6 by adding either Hydrochloric Acid (HCl) or Sodium Hydroxide (NaOH). Det här är formatet ”Text The titration endpoint information, combined with the other analyses conducted by the laboratory (major anions, cations and often elements such as Al, Fe, F and Si) made it possible to use Visual Minteq to model the charge balance.

The organic acid dissociation properties were determined from the discrepancy between the charge balance of measured anions and cations (Eq. 1). This discrepancy is largely due to the dissociation of organic acids. DOM concentration, alkalinity titrations and total aluminum and iron are other factors that will go into the analysis. At the pH 5.6 endpoint of the alkalinity/acidity titration performed on samples in the national surface water monitoring programs, the many components of the charge balance are most precisely determined from the information that can be retrieved from the MVM database

Eq. 1. Charge difference

$$\text{Charge difference (\%)} = 100 \times \left|\frac{Sum\ of\ Anions - Sum\ of\ Cations}{Sum\ of\ Anions + Sum\ of\ Cations}\right|$$

For the *Sum of Cations* the charge of the following elements and their complexes were used: Alx+, Cu3+, Zn, K+, Ca2+, Na+, Mg2+, Fex+, Si+, Znx+. For the *Sum of Anions* organic acids (OA-), SO42-, NO3-, Cl-, F- and their potential complexes where taken into account.

The charge contribution of different chemical species, both organic matter, but also Al, Fe and Si were determined by Visual Minteq. Visual Minteq's equilibrium chemical speciation modeling identified the state of different weak acid/bases (e.g. silica) and even complexation of Al with organic matter.

This model treats organic acidity as a set of organic acids with a set of fixed organic acid dissociation values. There is a parameter to determine how many organic acids a milligram of DOM has, termed the "Active DOM per DOC" (ADOM/DOC). By sweeping through a range of possible values for (ADOM/DOC) parameter in each sample, different Organic Matter Charge Densities at 5.6 in meq of charge per mg of carbon ($OMCD_{5.6}$) were calculated. The value of $OMCD_{5.6}$ that came closest to electroneutrality (i.e. which minimized the difference between positive and negative charge in the water sample) was selected as the $OMCD_{5.6}$ for that sample. If the minimum difference in charge balance was larger than 0.5%, then the sample was rejected as not having been satisfactorily modeled and excluded from further consideration in this study.

Patterns of $OMCD_{5.6}$ in space (between streams) and over time (for the same stream), as well as in relation to season and streamflow rates were also be explored, using national data resources. For the temporal analyses, the data were divided into three periods, 1990-1997, 1998-2011 and 2012-2024. The boundaries between the three time periods were determined to separate periods of more widespread change in the sVISa and TOC of the water at the watercourse sampling stations (cf. Fig. 1). The parameter sVISa is the specific visual absorbance, and is calculated from two parameters routinely measured by national monitoring in Swedish surface waters, the Absorbance at 420 nm and the DOM as:

Eq. 2. sVISa

$$sVISa = \frac{Absorbance\ filtered\ 420mm\ at\ 5cm\ depth}{DOC\ mg/l}$$

It is similar to SUVA, which uses Absorbance at 254mm instead of 420mm. The sVISa is used as an indicator of the character of DOM.

2.1 Determination of charge density

The following steps were performed to determine the charge density of organic matter ($OMCD_{5.6}$ meq of charge per g of carbon).

1. Raw water chemistry was downloaded from the MVM database for all streams in the database that had at least 10 years of continuous monthly sampling of TOC in the period 1990-2023.

2. These water chemistry data were processed to aggregate different measurement methods for the same variable into single columns. Here samples that did not have enough other parameters measured that were needed in the following steps were dropped from the analysis. Which parameters were deemed necessary is further elaborated in the Visual Minteq section of the methods.

3. The $OMCD_{5.6}$ was calculated by using Visual Minteq to balance the charge difference in the water sample at the titration endpoint of 5.6 across a range of ADOM/DOC values. The range of ADOM/DOC explored for each sample was from 0.05 to 3.5 in 0.05 intervals. Sjöstedt et al. 2010 used an ADOM/DOC value of 1.65 when modeling 322 samples of lake water from across Sweden.

4. Processing of Visual Minteq output to find the ADOM/DOC that best models the electroneutrality for each sample with the smallest charge difference. This “best fit” value of ADOM/DOC was used to calculate the $OMCD_{5.6}$ for each sample.

Once the $OMCD_{5.6}$ for all data were determined, the spatial and temporal variation of $OMCD_{5.6}$ across the dataset was explored statistically using the characteristics of the catchments upstream from each station where water chemistry was monitored.

2.2 Data sources

114 320 samples were dowloaded from the MVM Database (miljodata.slu.se/MVM/). Stations were preselected for streams that had TOC sampled 10 times a year for at least 10 consecutive years since 1990. Stations with

more than 5% urban area were excluded. This resulted in the download of data for 316 stations. Of these, 136 were used in the end as they

1) all the data required for the modeling,

2) these data had been analyzed by the SLU Department of Aquatic Sciences and Assessment Geochemistry Lab and

3) the charge balance could be closed sufficiently well by the Visual Minteq model.

The location, specific discharge, annual precipitation and mean temperature for these 136 stations are mapped on Figure 2.

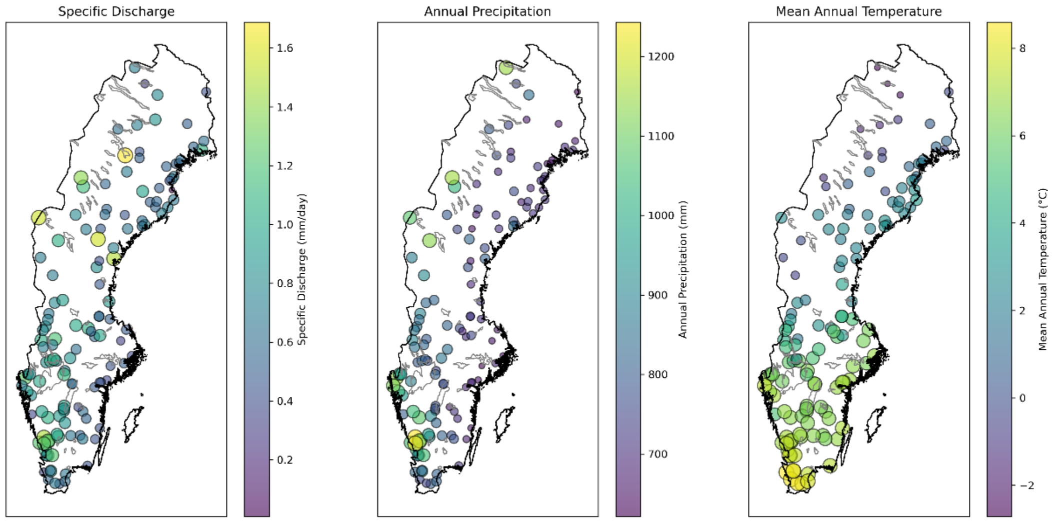


Figure 2. The 136 stations that were successfully modelled in this study. The size and color shows the mean specific discharge (left), the annual precipitation (middle), and the annual mean temperature (right) of the catchment for each station.

2.3 Preprocessing

- 42 907 samples across 187 stations meet the requirements for Visual Minteq.
- Median number of samples per station: 208 (cf. Appendix Fig A3 for the distribution of samples across the stations.)

- All samples that were not positively indicated as having been analyzed in the SLU lab were excluded. (If a sample entry in the MVM database did not have the “analysis lab” field filled in, these were excluded.)

- For further details about the modeling with Visual Minteq, see: Appendix 7.1 Visual Minteq Required parameters and Model set up

2.4 Post Processing

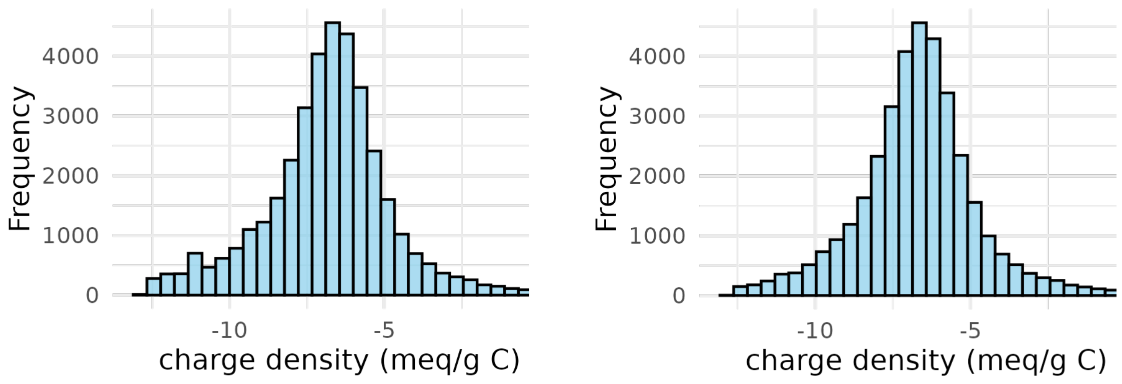
For each sample, the model run with the ADOM/DOC value that had the minimum absolute value of charge difference was selected as the best representation of the organic acid dissociation in that water sample at pH 5.6 (Appendix Fig. A4 for the almost 38,000 ADOC/DOC values) Charge difference was calculated according to Visual Minteq as Eq. 1. 9

Ideally, this charge difference should be zero, but since the optimization of the ADOC/DOC value went through a range of possible ADOC/DOC values (0.05 to 3.5) in 0.05 intervals, the closure of the charge balance was not exact. If the smallest charge difference % that could be achieved was greater than 0.5%, the water sample was excluded from further analysis as not sufficiently well modelled. This left just over 36,000 satisfactorily modeled water samples from 136 stations. These had an approximately normal distribution (Fig. 3), but ca 2,000 samples (just over 1% of the acceptably modeled samples at the minimum value of the range explored).

Once the optimal ADOM/DOC was determined for each sample, the corresponding organic charge was used to calculate the charge density ($OMCD_{5.6}$) using best fit ADOM/DOC for each sample and the concentration of DOM of the sample.

Eq. 3. Charge density

$$\text{Charge density (meq/g C)} = \left|\frac{\text{organic charge (meq/l)}}{\text{TOC (mg/l) / 1000}}\right|$$



(a) Before filtering of charge difference < 0.5% (b) After filtering of charge difference < 0.5%

Figure 3. Histogram of charge density for samples before (a) and after filtering (b) out the samples with a charge difference larger than 0.5%.

Data Normality: To assist in choosing the statistical methods to analyze the differences between stations and between periods, the median values of observed and modeled chemical parameters from the different stations (Appendix Table 2, Appendix Figure A10). Normality was evaluated, with and without log transformations. Based on these evaluations, non-parametric statistical tests were adopted.

2.5 Catchment Characteristics

Soil Depth, Land Use, Climate, and Runoff: To explore factors that correlate to charge density ($OMCD_{5.6}$) in space and time, data from the study catchments was harvested. The Soil Depth was collected from the SGU Soil Depth Raster Map (https://www.sgu.se/produkter-och-tjanster/kartor/kartvisaren/jordkartvisare/jorddjup/). Land Use Categories came from the National Soil Data map (Naturvårdsverket, 2018), climate information came from SMHI's gridded data (smhi.se/data/ladda-ner-data/griddade-nederbord-och-temperaturdata-pthbvand runoff data from SMHI's S-Hype Model (smhi.se/data/hydrologi/Vattenwebb). The climate of the study sites varied in terms of mean annual temperature (-3 to 7 °C) and precipitation (600-to almost 1200 mm) (Appendix Fig. A5). When dividing the available water chemistry data for this study into three periods (1990-1997, 1998-2011 and 2012-2024), the mean annual temperature was not significantly different between the periods. The precipitation however, was significantly higher in the middle period, relative to both the first and third periods. (Appendix Figure A6)

Area proportional annual discharge was calculated for each water chemistry sampling station (Appendix Fig. A7), using the local discharge when considering sub-catchments of the SMHI unit catchments (Aroid catchments). When the SMHI subcatchment is smaller than the catchment of the sampling station, the discharge from the entire Aroid catchment was used. Where possible upstream station corrected discharge was used. Discharge is in m3/s and specific discharge in mm/day.

NDVI: As a measure of primary productivity by vegetation in the landscape, the Landsat Normalized Difference Vegetation Index (NDVI) was used. These values were extracted using the Google Earth Engine for monthly time series for each catchment of median NDVI from the LandSat NDVI images (Appendix Fig. A8). These are taken at eight-day intervals during the study period (1990 -2024) using four different Landsat satellites. These data were preprocessed and aligned between the different satellites. The NDVI values increased steadily and significantly during the three study periods (Fig. 4).

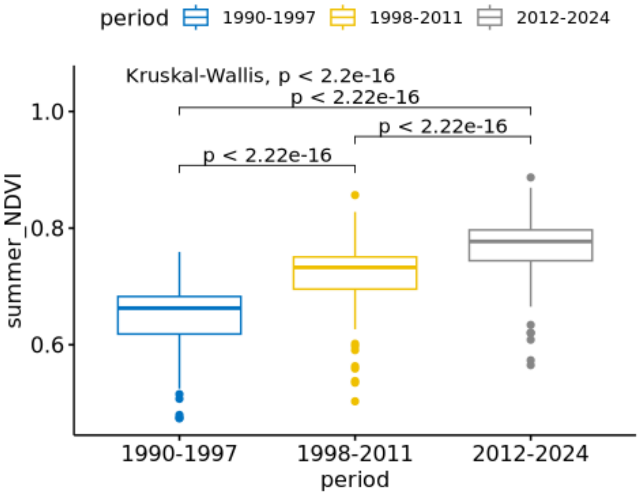


Figure 4: The Summer NDVI June-August for all catchments in the study increased with time between the periods 1990-1997, 1998-2011, 2012-2024.

Peat: The area of peat is of particular concern for studies on DOM. SLU has worked with SGU, SMHI and the Swedish Mapping, Cadastral and Land Registration Authority (Lantmäteriet), along with original data from the national soil survey to compile a comprehensive map of areas where peat is more than 30 cm deep (https://www.slu.se/en/departments/forest-ecology-management/forskning/torvkartor/here-are-the-maps/) . This was used to define the peat area in all catchments of this study (Appendix Figure A9).

A summary of the catchment information used by this study is found in Appendix Table A2. Most values are given as the percentage of the area in the catchment covered by the specific data source. Exceptions are:

- Temperature i.e. MAT (mean annual average in °C)
- Discharge (annual average and interquartile range of the averages m3 s-1)
- Precipitation (mm/year), summer NDVI (NDVI for mean of June July and August for every year averaged over the time period 1990-2023)

Specific discharge mean, p25, p75, (mm d-1). (This is median, p75, p25 for extracted for each year and then averaged over the entire period.)

3. Results

3.1 Water chemistry variation: measured and modeled station comparison

Just over 36,000 water chemistry samples from 136 watercourse monitoring stations between 1990 and 2024 were successfully modeled using Visual Minteq to determine the $OMCD_{5.6}$. There is considerable variation between the median TOC and sVISa values of Swedish water courses, and for the individual values within each monitoring station (Fig. 6). This is already relatively well known. What has not been quantified before is the variation in $OMCD_{5.6}$ (Fig 6 c). For $OMCD_{5.6}$, the median of the station medians was – 6.7 meq g^{-1} C, with an IQR between the 25th and 75th percentile stations of 2. The extreme values of the station medians were -8.9 and -4.0 (Appendix Table A2).

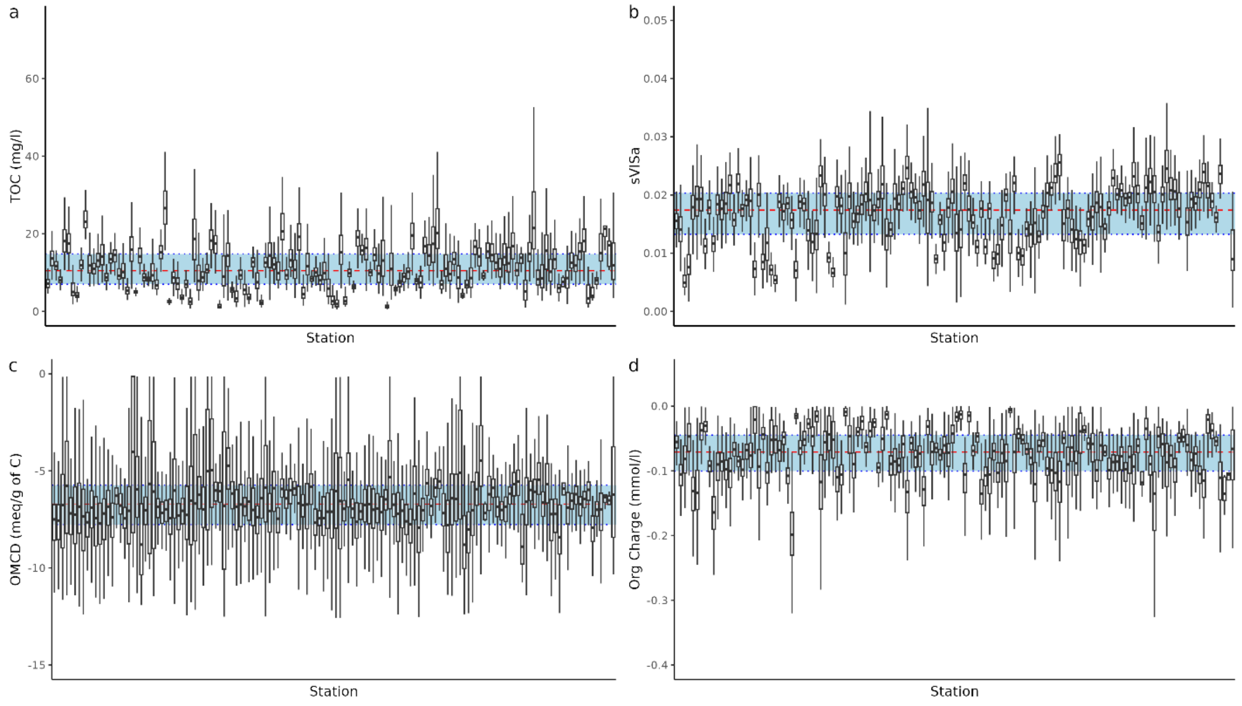


Figure 6: Boxplot for each of the stations sample distribution of TOC (a), sVISa (b), $OMCD_{5.6}$ (c.), and organic charge (d.). The blue box is the weighted IQR for all the stations, with the red dashed line as the weighted median for all the stations.

It is interesting to note if there are correlations between the $OMCD_{5.6}$ and any other chemical parameters (Appendix Fig A11). There was a small degree of positive correlation with the total amount of organic charge, and a small negative relation to pH and alkalinity. Otherwise $OMCD_{5.6}$ had little correlation with water chemistry.

3.2 Temporal Patterns

To evaluate changes over time, the data from each individual catchment was split into three periods, 1990-1997, 1998-2012 and 2013-2024 (Appendix Table A4, Appendix Fig. A13). The changes over time were accompanied by changes in the contribution of strong and weak acids, as well as the organic acids contribution (Fig. 7). The changes in $OMCD_{5.6}$ is included in Fig. 7 for comparison. It should also be noted that the number of stations included in each period varied, and the number of samples per station varied between the different periods (Appendix Fig. A12).

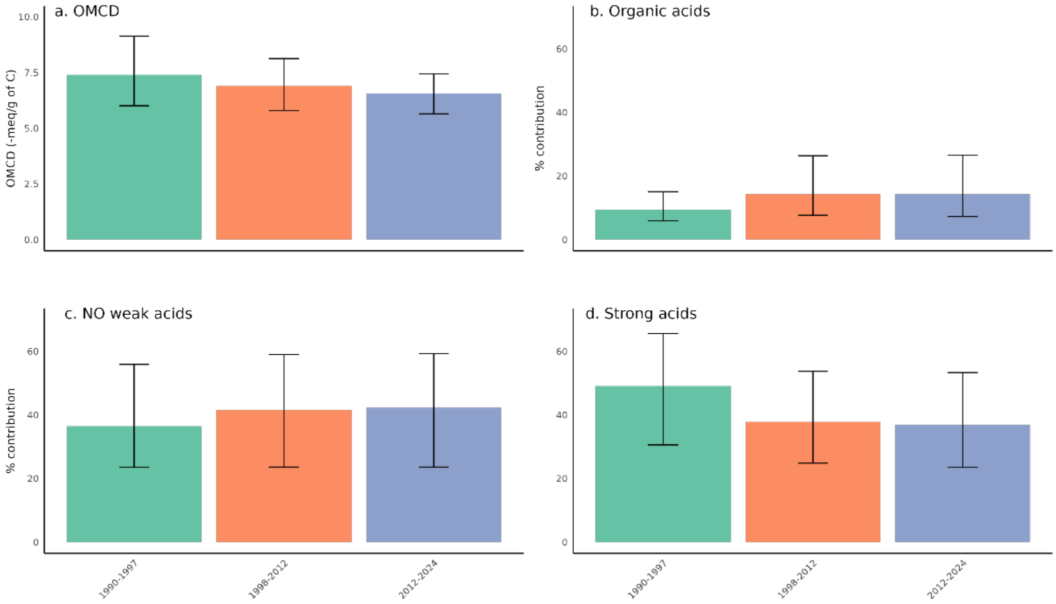


Figure 7: Bar graphs of (a) $OMCD_{5.6}$, and the percentage contribution to charge in the water samples from (b) organic acid, (c) non-organic weak acids and (d) strong acids. This is presented for the three periods, with all stations weighted by the number of samples in each station. The percentage contribution is calculated as the percentage of the total negative charge.

For each station, changes in the water chemistry between the three periods, 1990-1997, 1998-2012 and 2013-2024 were tested using Kruskal-Wallis, providing there were at least two years of samples in each period. Significant differences between the periods were found for a number of parameters, for both measured and modeled values, including sVISa, TOC (Appendix Tables A5 and Appendix Fig. A14, A16, A18) and $OMCD_{5.6}$. Of the 139 stations evaluated, more than half had significant changes between one pair of the three periods. For sVISa, 80 stations had a significant change, for TOC it was 67 stations, and for $OMCD_{5.6}$ 77 stations (Fig. 8 and Appendix Table A5).

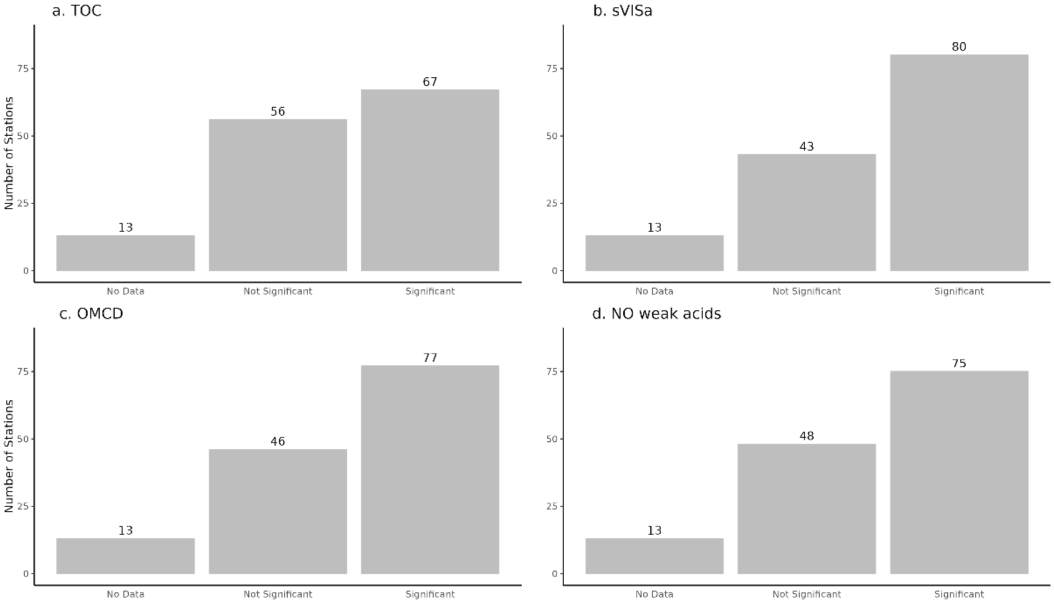


Figure 8: Number of stations with significant differences between at least two periods as found by a Kruskal Wallace test with $p < 0.05$ for TOC (a), sVISa (b), $OMCD_{5.6}$ (c), and the contribution of NO weak acids (c).

N.B. "Positive change" in $OMCD_{5.6}$ means that the charge density became less negative since organic charge is negative. Thus, the "increase" in $OMCD_{5.6}$ means that the organic matter became less charged.

For each station that had a significant change between any of the three possible pairs of periods (1990-1997 vs 1998-2012; 1990-1997 vs 2013-2024; 1998-2012 vs 2013-2024), Lasagna plots stacked vertically by latitude indicate if there was a change in sVISa (Appendix A14), TOC (Appendix A16) and $OMCD_{5.6}$ (Fig 9). The strong positive change in $OMCD_{5.6}$ between the first period and the third period is evident. Two things to bear in mind, however, are that 59 of the 136 stations did not have a significant change in $OMCD_{5.6}$. The "positive change" in $OMCD_{5.6}$ also means that the charge density decreased, since organic charge is negative, and an increase in the value means that this value became less negative. This would suggest that the organic matter is becoming less soluble in water.

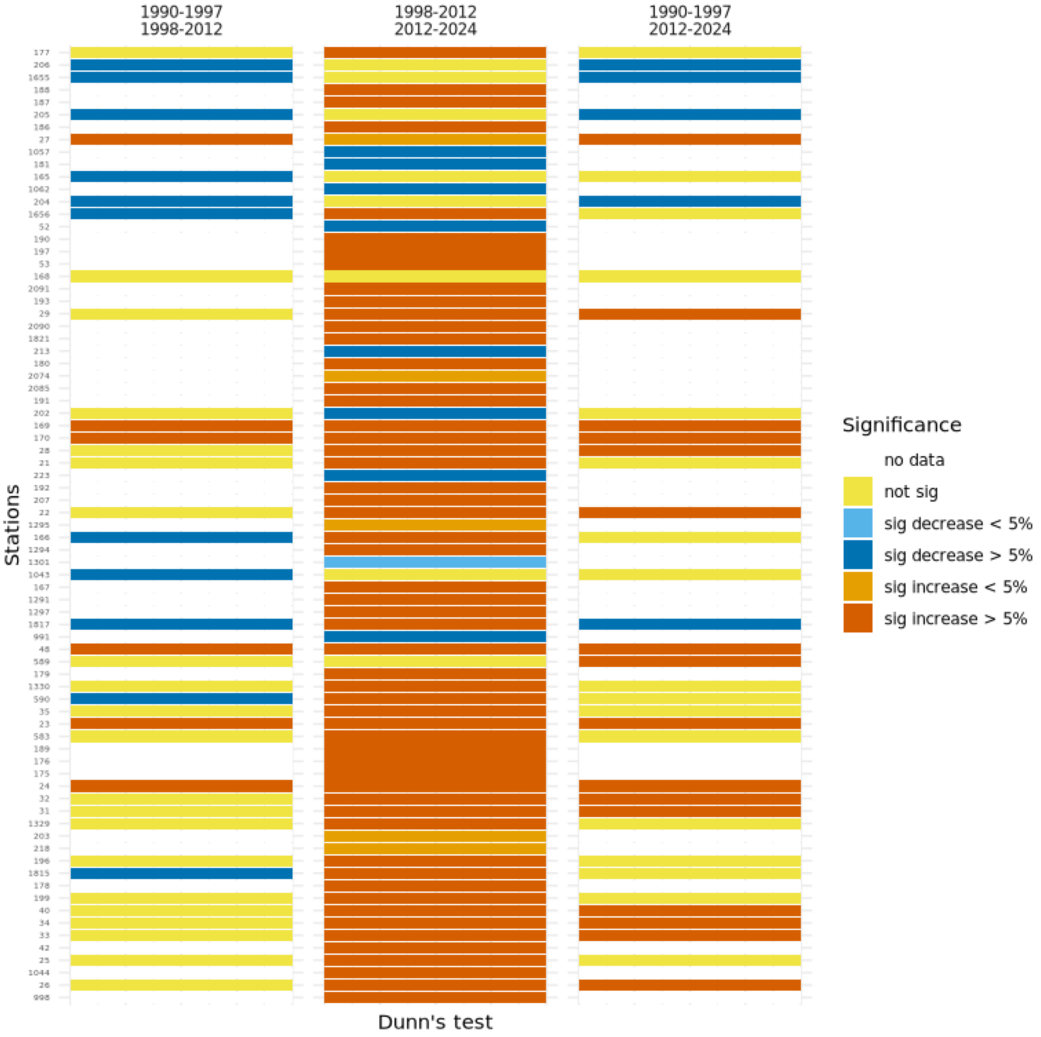


Figure 9: Organic charge density changes between periods. Only stations for which Kruskal-Wallis test found a significant difference between periods are included.

N.B. "Positive change" in $OMCD_{5.6}$ means that the charge density became less negative since organic charge is negative. That the "increase" in $OMCD_{5.6}$ means that the organic matter became less charged.

Looking at the median $OMCD_{5.6}$ of all the stations, the decrease in how negatively charged TOC becomes over time is also evident, with median station $OMCD_{5.6}$ going from -7.4 in the first period, to -6.9 in the second period and then -6.6 in the last period (Fig. 11 and Appendix Table A4). Similar changes in overall station medians are noted for sVISa and TOC, with the largest increase in sVISa and TOC occurring when comparing the first and third periods (Appendix Figures A15 and A17, Appendix Table A5).

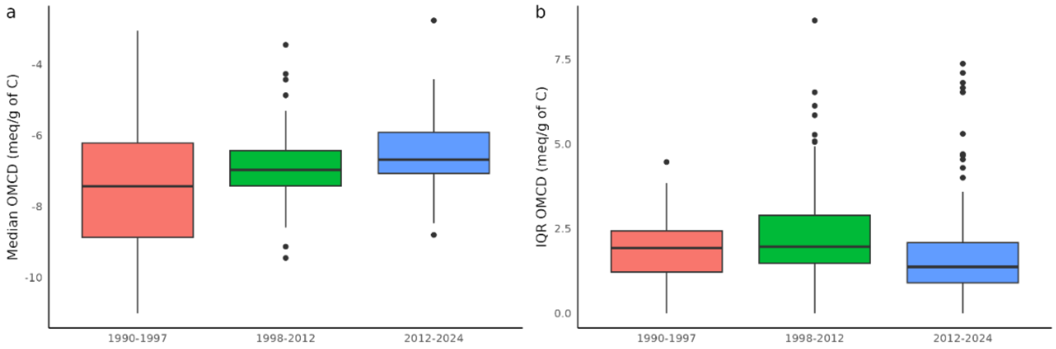


Figure 11: OMCD$_{5.6}$ medians (a) and IQR (b) of all stations.

A final aspect of how the water chemistry has changed over time is the contribution of the organic matter to the acidity of water. The acids in a water chemistry sample can be split into weak acids and strong acids. The weak acids can be further divided into organic acids and other weak acids (Table 1).

The weak acid contribution is calculated:

Eq. 4. Charge contribution

$$\begin{matrix}\text{charge contribution of} \\ \text{non-organic weak anions}\end{matrix} = 100 * \frac{\text{Sum of anions} - (\text{Organic anions} + \text{Sum of strong anions})}{\text{Sum of anions}}$$

Here we look at how the contribution of these other weak (non-organic) acids has changed over time for each station. There were significant changes in this value between at least one pair of the three periods for 77 stations (Appendix Table A5). When there was a change, it was generally an increase in the contribution of non-organic weak acids (Appendix Fig. A18). When considering the station median contribution of non-organic weak acids, one sees little change (Fig. 11).

Here we look at how the contribution of these other weak (non-organic) acids has changed over time for each station. There were significant changes in this value between at least one pair of the three periods for 77 stations (Appendix Table A5). When there was a change, it was generally an increase in the contribution of non-organic weak acids (Appendix Fig. A18). When considering the station median contribution of non-organic weak acids, one sees little change (Fig. 11).

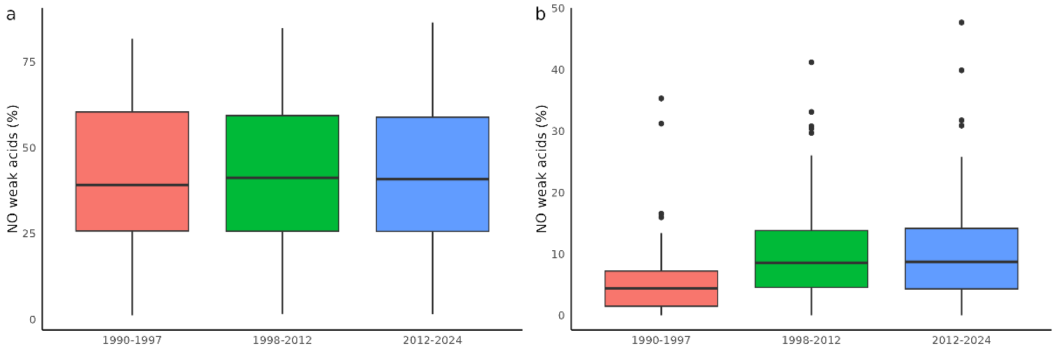


Figure 11: NO weak acid contribution (%) medians (a) and IQR (b) of all stations.

Table 1: The components of the sum of anions for each water chemistry sample, its constituents and whether it is modelled or measured.

Anion Component	Constituents	source
strong acids	SO4, Cl, NO3	measured
not organic weak Acids	Al, Fl, etc….	modelled
organic acids	DOC	modelled

3.3 Spatial Patterns

When looking at the mean values of sVISa, TOC and $OMDC_{5.6}$ across Sweden, there are no readily apparent patterns (Fig. 12). To see which catchment characteristics were related to the amount and character of TOC, Orthogonal Partial Least Squares (OPLS) was used to separate the variation correlated (predictive) to the factor of interest and the uncorrelated (orthogonal) variation. This works similarly to the more commonly used PLS, but OPLS facilitates interpretation. The R package ropls was used for these analyses. The underlying code of this package is that of the SIMCA software. The explanatory variables used in the OPLS are the catchment characteristics listed in Appendix Table A2, and the detailed results of the OPLS results are found in Appendix Table A6.

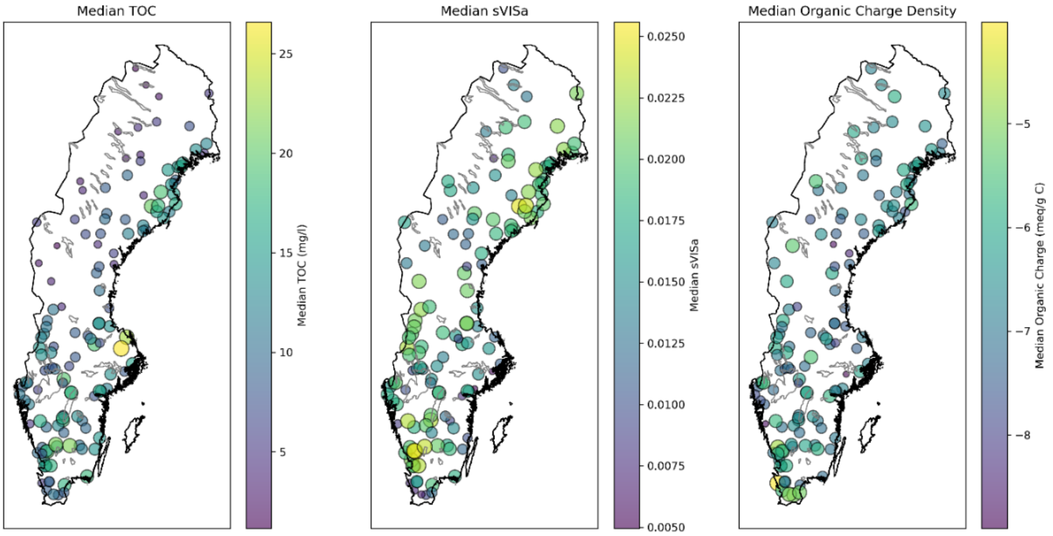


Figure 12: Water chemistry modelled and observed for 136 stations with available data that were successfully modelled in this study. The size and the color shows the observed median TOC (left), the observed sVISa (middle), and the modelled organic charge density (right) of the catchment of the station.

As other studies of Swedish surface waters have found, the concentration of TOC is positively related to the amount of peat, forest, mean annual temperature and summer NDVI. Negative influences on TOC included area of mineral soil and elevation (Appendix Fig. A20 b). Several of the factors with a positive correlation to sVISa were the same as for TOC (e.g. peatland, forest on wetlands). Negative influences that were not present for TOC, but appear for sVISa, are the area of lakes/watercourses in the catchment, catchment area, and the log of mean annual discharge.

For the $OMCD_{5.6}$, a somewhat different set of positive catchment influences are evident (Fig. 13). Precipitation, elevation and specific discharge are positively related, along with hedmark (heathland). Several negative influences related to residence time of surface water (catchment area, area of lakes/watercourses).

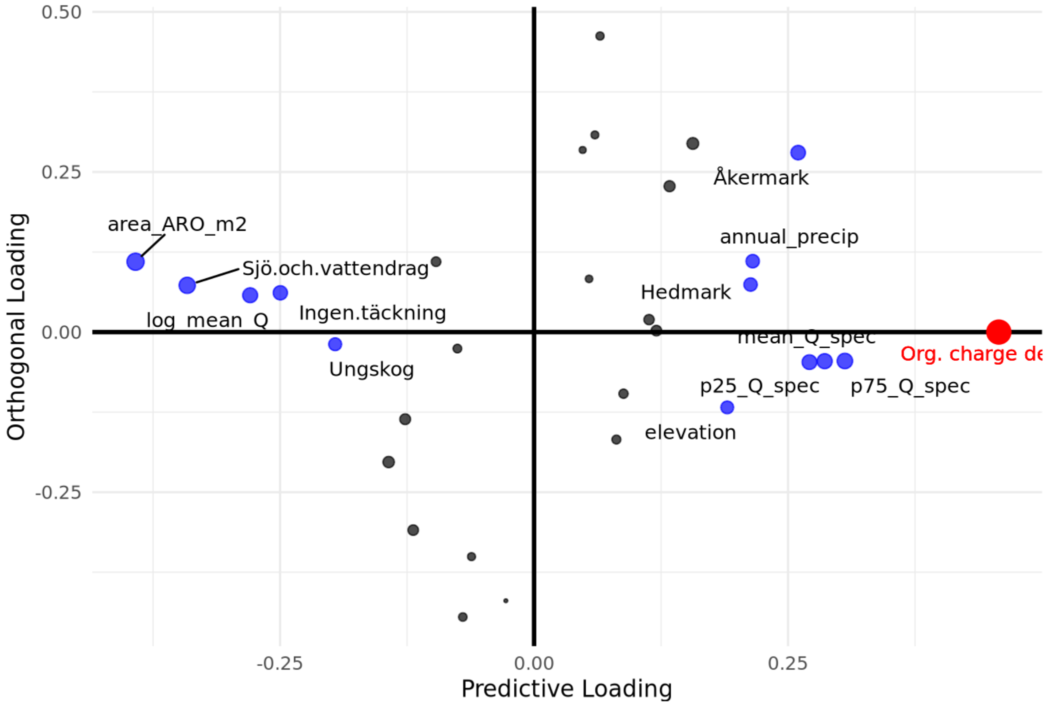


Figure 13: OPLS loading plot for Organic charge density. Blue variables indicate a VIP score > 1 while black indicate a VIP < 1. The red is the response variable: organic charge density. The x-axis is the predictive axis indicating covariation with the response while the orthogonal axis is variation not explaining any variation in the response variable.

4. Discussion

While acid deposition has declined, there are still many relatively acid streams and lakes in Sweden. Some of this acidity arises from dissolved organic matter (DOM). Targeting acidification countermeasures often including quantification of the natural acidity provided by DOM. This involves determining both the natural concentration of DOM, and calculating how DOM influences pH. Changing DOM concentrations across Sweden in recent decades has received attention in this regard (e.g. Erlandsson et al, 2011), since both reduced acid deposition, forest management and drainage can increase DOM (Kritzberg et al., 2019). Less attention, however, has been paid to whether the acid-base properties of DOM have been changing. The dissociation of DOM, as measured by the charge density at any given pH, is critical for determining the influence of DOM on pH and the assessment of natural acidity. The charge density is also related to the solubility of soil organic matter. Since changes in the character of SOM can be inferred from DOM (Tian et al., 2025), the changing DOM dissociation properties of organic matter identified in this study can also be a factor in the "brownification of waters".

The possibility of these dissociation properties changing is generally not considered in acidification assessments, even though another feature of DOM character has been changing in many watercourses, namely sVISa, the relation between DOM and water color (Eklöf et al., 2021).

The use of uniform DOM dissociation properties seemed justified when methods for this were established during the 1990s since these DOM dissociation properties were relatively uniform in both heavily acidified and pristine areas (Hruska et al 2003). Only several years of data were available at that time though. Now there are tens of thousands of lake and stream chemistry measurements from across Sweden collected over more than three decades. While it has generally required specialized studies of a relatively small number of water samples to determine the acid-base dissociation properties of DOM, the present study has succeeded in using the data in the MVM database to determine one feature of organic acid dissociation in almost 40,000 water samples collected from 136 national surface water monitoring stations between 1990 and 2024. That feature is the density of negative of charge per gram of dissolved organic matter at a pH of 5.6 ($OMCD_{5.6}$). (pH 5.6 is the endpoint of the acid-base titration conducted on water samples in the national surface water monitoring programs.)

This study has quantified the variation in TOC, sVISa, organic charge and $OMCD_{5.6}$ and total organic charge within a given station and between stations (Fig. 6). $OMCD_{5.6}$ stands out by having less variation between station medians, while having a larger variation within each station. This is evident from how the station medians for $OMCD_{5.6}$ generally fall within the IQR of the station medians.

The spatial variation of $OMCD_{5.6}$ (i.e. differences between stations) is weakly related to the acidity of water, and several features of catchments (Fig A11, Fig 13). For instance, peat soils are associated with somewhat greater charge density. On the other hand, the area of lakes/water course or catchment size reduces the charge density (Table A6). That latter correlation might be a result of light exposure breaking down organic acid functional groups.

Over time, the median station $OMCD_{5.6}$ has changed in two thirds of the stations, with the direction of change more often positive than negative. The "increase" in $OMCD_{5.6}$ means that the organic matter became less charged, since organic matter is negatively charged. This decline in charge density would make a given amount of TOC lower the pH in a water sample less, presuming this decrease in charge density occurs across the full range of pH found in Swedish surface waters. A lower charge density in more recent years would also suggest a lower solubility of soil organic matter, since it is the negative charge of OM that contributes to OM solubility.

The direction of change in $OMCD_{5.6}$ found by this study (less charge density), was different than that found by Vogt et al. (2024), who found that charge density not only increased, but also to a larger degree than our study found. The study region and methodologies of that study differs from this study, so the reasons for the differences will need to be analyzed for comparability.

So how important is the variation and changes in charge density? While it is interesting to find variation in one feature of the acid base properties of DOM, this begs the question of how important this variation is for the pH of surface waters. The answer to this question varies from site to site, since the influence of a given quantity of negative charge on pH relates to where on the pH range a water sample is, and the amount of DOM in the sample. For surface water at pH 5.6, one µeq of negative charge changes pH much more than at pH 4.4, due to the exponential nature of the pH sale.

To provide an initial estimate of the significance of the observed variation in $OMCD_{5.6}$, we compare the amount of variation in negative charge (equivalents) that organic matter provides due to variation in $OMCD_{5.6}$, relative to the total amount of charge that organic matter provides. Taking the mean TOC concentration and median organic charge density of all water samples in this study, we get a median organic charge of -70 µeq/l. The standard deviation of organic charge density, multiplied by the median TOC concentration yields a standard deviation of organic charge of 21 µeq/l. This means that, as a first approximation, the variation of the dissociation properties changes the contribution of organic acids to the charge balance by +/- 30%.

It will take more detailed analyses to reveal what this means for pH in specific cases, but at this point, we can say that this degree of variation in the organic acid dissociation between water samples at the same pH is not consistent with the assumption that spatial and temporal variation in the organic acid dissociation properties of TOC do not need to be accounted for when assessing the role of organic acids in surface water chemistry. A key assumption in making that statement, however, is that this variation in $OMCD_{5.6}$ and total organic charge is modeled with as much precision as the values of TOC and sVISa can be measured. That is an assumption that bears looking at more closely.

Several other features that deserve further attention with respect to the changes over time reported in this study are how the number of stations providing data, and the measurement of certain trace elements varied. The number of stations included in each period varied, and the number of samples per station varied between the different periods (Fig. A12). The number of samples where certain trace chemical elements were analyzed also varied between periods. Our initial examination of possible influences have not suggested that these changes influenced the results. For instance in the contribution of non-organic weak acids, and the proportion of weak acids (Fig. A19). More however, could be done to check for eventual influences. One way to do this would be to save more of the Visual Minteq results to document the charge contributions from silica and fluoride.

Perhaps the most important feature of this study that needs borne in mind is that the systematic quantification of the degree of organic acid dissociation, and the associated charge density is available at only a single pH value, 5.6. The dissociation of organic acids, however is something that changes over the entire range of pH in surface waters. The limitation to a single pH is due to the information that is saved from the routine titration of water chemistry in the national monitoring program. In principle, the dissociation of the organic acids could be determined at a number of pH's from this titration. However, the titration is optimized to give only a reliable pH at the titration endpoint, and this endpoint, along with the amount of acid/base needed to reach that endpoint, is the only information saved in the MVM database. By simply saving more information from these titrations, such as the pH before and after the CO_2 is degassed at the start of the titration, a better picture of the dissociation of organic acids over the natural range of surface water pH could be adduced. The value of these titrations could be further enhanced in a special study by optimizing the titrations for the purposed of determining organic acid dissociation.

5. Conclusion

Acidification classification has been questioned with regards to how DOM influence on acidity is handled (HVMFS, 2013). This project has leveraged the national monitoring data to address this area of uncertainty. This is of special importance since there is a new national liming strategy in preparation.

The findings of this study indicate that there is variation in the acid base properties of organic matter that could influence the contribution of DOM to surface water acidity. Further analysis, however, is needed to determine whether a more regionally and temporally nuanced characterization of organic acidity could allow SLU to contribute more to the national work on targeting liming for acidification mitigation. To be useful in this way, though, it will be necessary to explore the dissociation of organic acids across the natural range of pH in Swedish surface waters. This could be done with just the information from this study, but even more effectively if further information from the routine titrations of surface waters in national monitoring programs is collected.

6. Acknowledgments

This study was funded by a grant from SLU's Environmental Monitoring and Assessment Acidification Programme.

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8. Appendix

8.1 Visual Minteq Required parameters and Model set up

1. Silicon was set to same value as in Sjösted et al, 2010: 0.01mM when it was not measured.
2. Bold parameters had to be measured parameters in the raw data, other parameters were set to 0 if they were not measured.

Appendix Table A1

Name	Variable	Valance
Aluminium	Al_mol	
Copper	Cu_mol	+3
Manganese	Mn_mol	+2
TOC	TOC_mol	-x
Zinc	Zn_mol	
SO4	SO4_mol	-2
NO3	NO3_N_mol	-1
Cl	Cl_alk_mol	-1
Pottasium	K_mol	+1
Calcium	Ca	+2
Sodium	Na_acid_mol	+1
Magnesium	Mg	+2
Iron	Fe	+3
Silicon	Si	
Ammonium	NH4	+1
Fluoride	F	-1

8.1.1 Modelling Set-up

3. temperature set to 10°C
4. pH $_{5.6}$
5. Ferrihydrite as possible solid phase
6. AlOH3 as possible solid phase
7. ADOM/DOC sweep from 0.05 to 3.5
8. alkalinity/acidity was added as Na and Cl concentration respectively.

8.2 Supplementary Information


(a) 316 stations

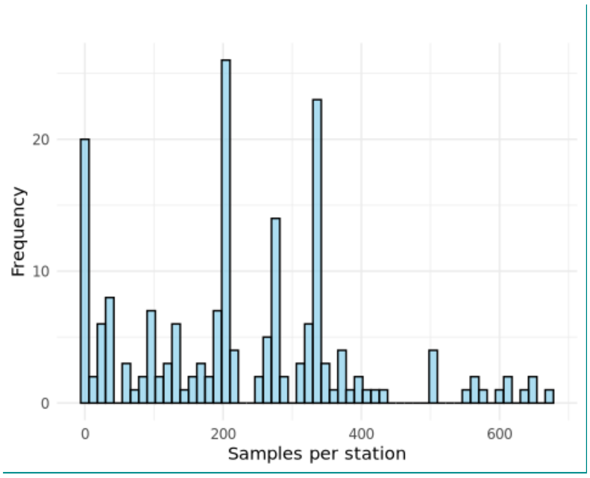
(b) 136 stations of this study

Appendix Figure A1: Lasagna plot of sVISa trends across Sweden. Updated from Eklöf et al, 2020. (a) All 316 stations that fit the initial selection criteria for this project of 10+ years of TOC data and < 5 percent urban area. (see Methods for more details on these criteria). (b) The 136 stations where charge density was modelled of this project (see Results for an explanation of which stations were modeled successfully).

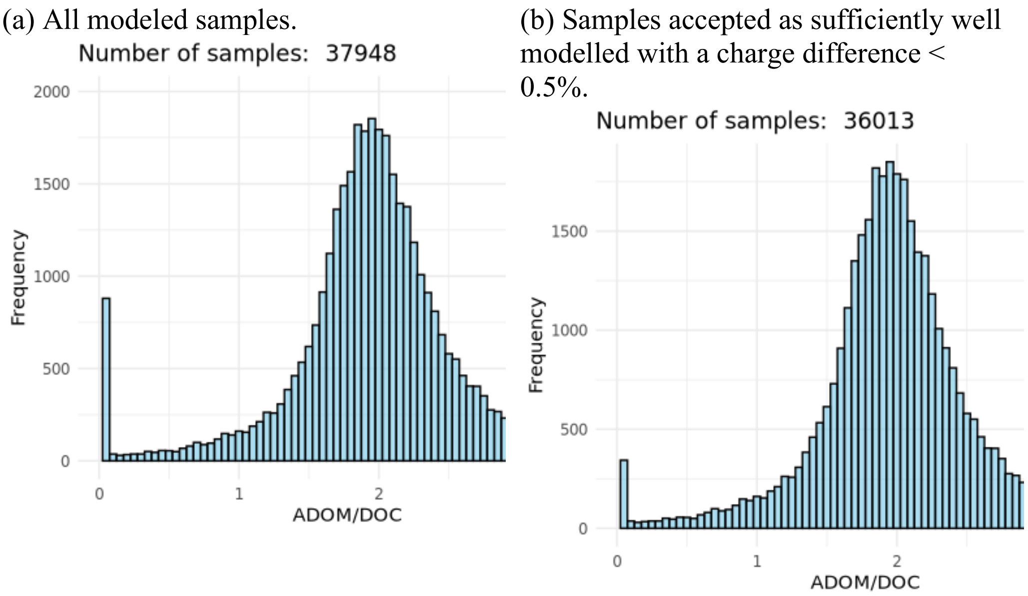
a) 316 stations

(b) 136 stations of this study

Appendix Figure A2: Lasagna plot of TOC trends across Sweden. Updated from Eklöf et al, 2020. (a) All 316 stations that fit the initial selection criteria for this project of 10+ years of TOC data and < 5 percent urban area. (see Methods for more details on these criteria). (b) The 136 stations where charge density was modelled of this project (see Results for an explanation of which stations were modeled successfully).

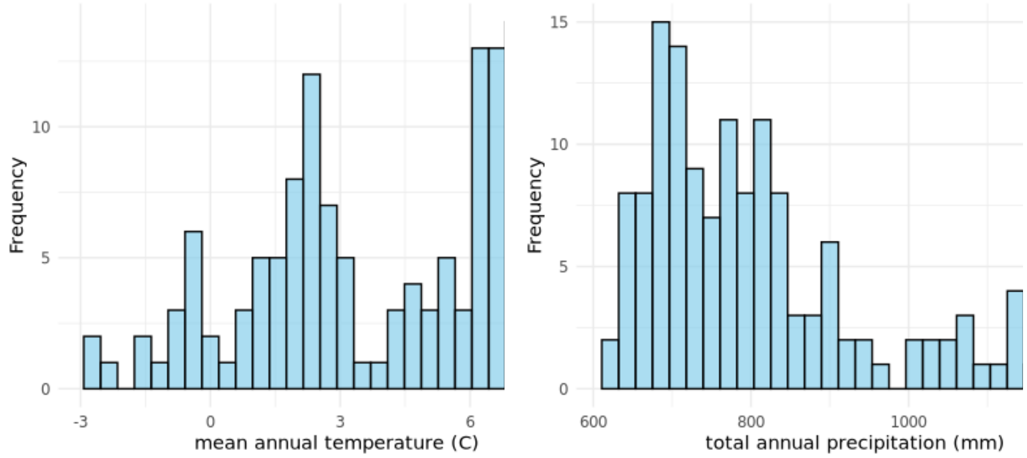


Appendix Figure A3: Histogram of the number of samples at different the stations that were retrieved from the MVM database. There are 42 907 samples across 187 stations. In further analysis only stations with more than 5 samples measured by the SLU laboratury were considered, reducing the number of stations to 136.


(a) All modeled samples.

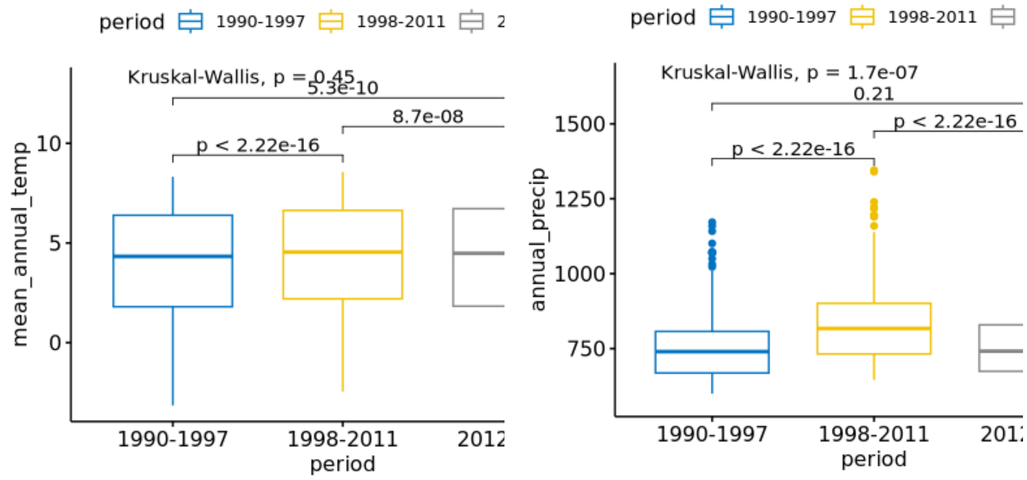
(b) Samples accepted as sufficiently well modelled with a charge difference < 0.5%.

Appendix Figure A4: Histogram of ADOM/DOC all modeled samples (a) and only samples accepted as sufficiently well modelled with a charge difference < 0.5% (b).



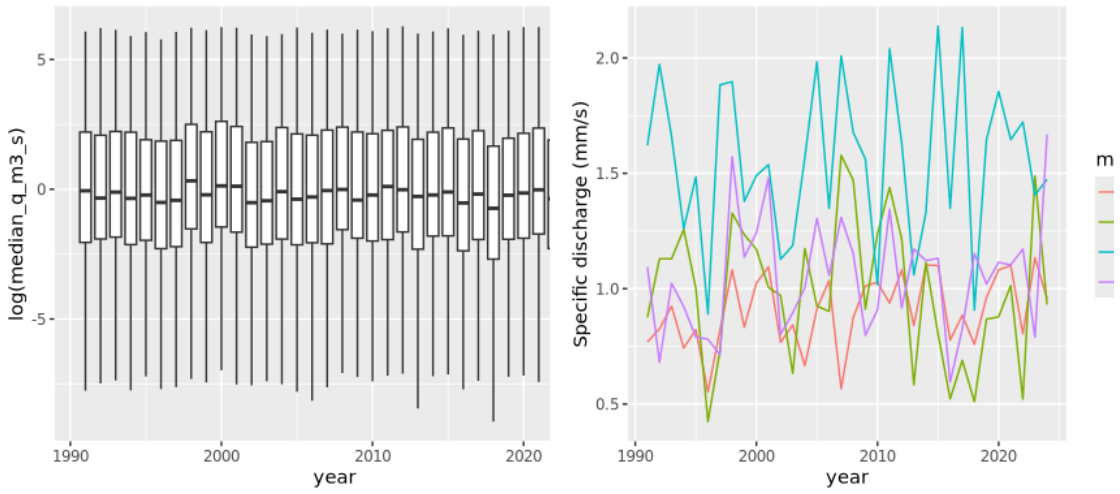
(a) mean annual temperature (b) total annual precipitation

Appendix Figure A5: Histogram of climate variables.



(a) Temperature (b) Precipitation

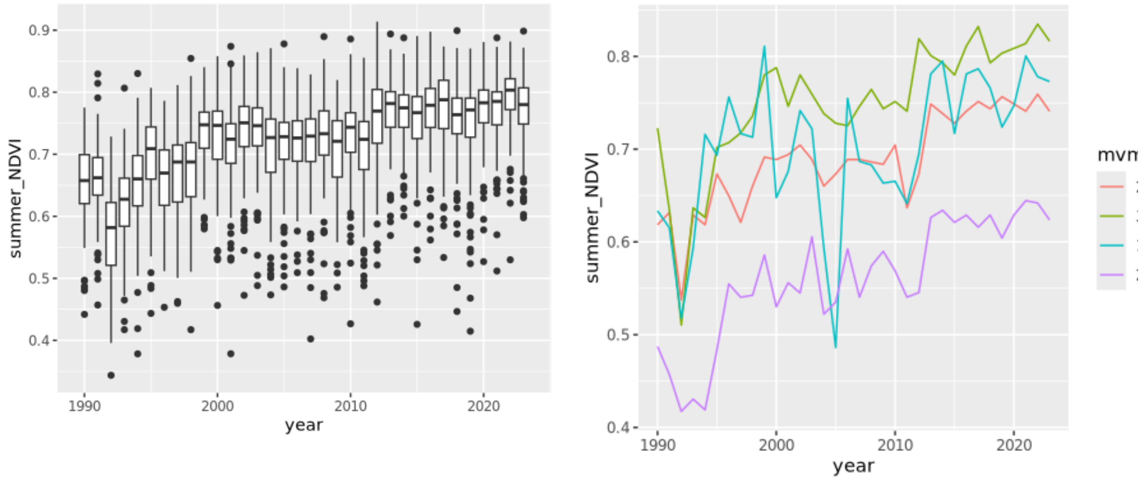
Appendix Figure A6: Boxplots of climate variables for the three periods 1990-1997, 1998-2011, 2012-2024.



(a) Boxplot of all the catchments together.

(b) Example of median annual discharge for 4 different catchments.

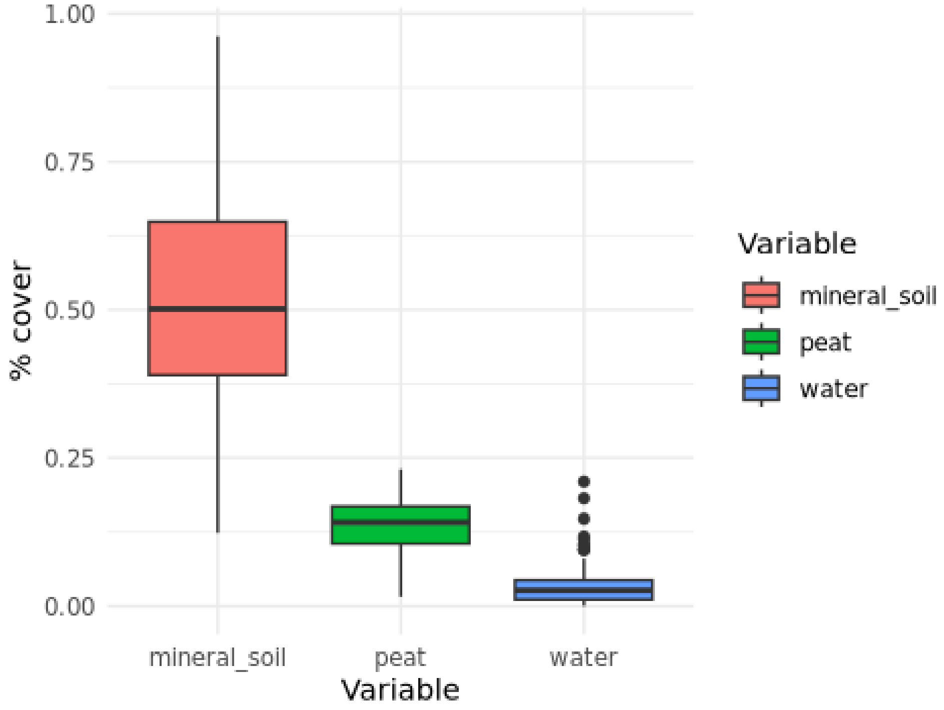
Appendix Figure A7: Discharge for all catchments in the DOC dissociation study.



(a) Boxplot of all the catchments together.

(b) Example of summer NDVI for 4 different catchments

Appendix Figure A8: Summer NDVI June-August for all catchments in the DOC dissociation study.



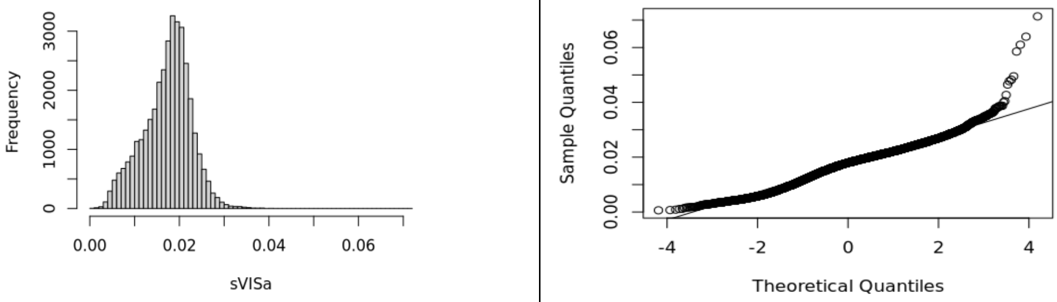
Appendix Figure A9: Peat area data from the SLU Peat Map.

Appendix Table A2: Catchment characteristics for the stations used in this study.

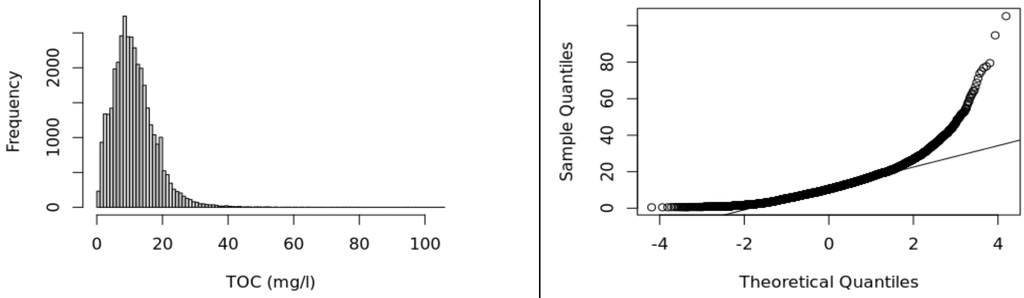
Variable	*N*	*Mean*	*Std. Dev.*	*Min*	*Pctl. 25*	*Pctl. 75*	*Max*
Ingen.täckning	122	1.47	7.13	0	0	0	61
Öppen.våtmark	136	8.07	7.82	0	2.65	10.5	41.7
Åkermark	136	5.13	11.6	0	0	4.73	81.5
Hällmark	136	1.12	4.14	0	0.0176	0.239	35.8
Hedmark	136	8.36	15.2	0	1.65	7.02	80.1
Hårdgjorda.ytor	136	0.116	0.168	0	0.00618	0.152	0.913
Semiurbant	136	1.93	1.24	0	1.09	2.9	4.45
Sjö.och.vattendrag	136	4.79	4.16	0	1.72	6.48	20.2
Tallskog	136	20.5	14.5	0	10.5	28.2	68.4
Gran_barrblandskog	136	18.5	12.8	0	10	23.6	95.3
Blandskog	136	5.56	4.44	0	2.33	7.87	21.4
Lövskog	136	6.14	8.72	0	1.81	7.47	77.4
Ungskog	136	13.2	9.63	0	8.54	18	84.9
Skog.på.våtmark	136	5.15	4.56	0	2.29	7.07	27.4
peat	125	0.136	0.0455	0.0157	0.105	0.168	0.231
mineral_soil	125	0.513	0.179	0.124	0.39	0.649	0.961
soil_depth_iqr	136	5.97	4.71	0	3	8	30
soil_depth_mean	136	8.09	6.13	0.0643	4.26	10.6	39.7
mean_annual_temp	136	4.01	2.91	-2.71	1.97	6.62	8.6
annual_precip	136	805	141	622	698	852	1243
summer_NDVI	136	0.718	0.0528	0.519	0.697	0.75	0.844
mean_Q_spec	136	1.07	3.74	0.0075	0.531	0.93	44.2
p25_Q_spec	136	0.58	2.29	0.00382	0.248	0.474	27
p75_Q_spec	136	1.98	6.45	0.0138	1.07	1.75	76.4
area_ km2	136	2513	6737	0	21	1119	48126
elevation	136	152	159	-0.1	18.5	204	704
log_mean_Q	136	0.108	2.96	-7.46	-1.92	2.16	6.1

Appendix Table A3: Water chemistry values measured and modelled for the stations used in this study. The values from each station were weighted proportionally to the number of samples for each station for the Median and IQR while Min Median and Max Median refer the median for the station with the minimum/maximum median.

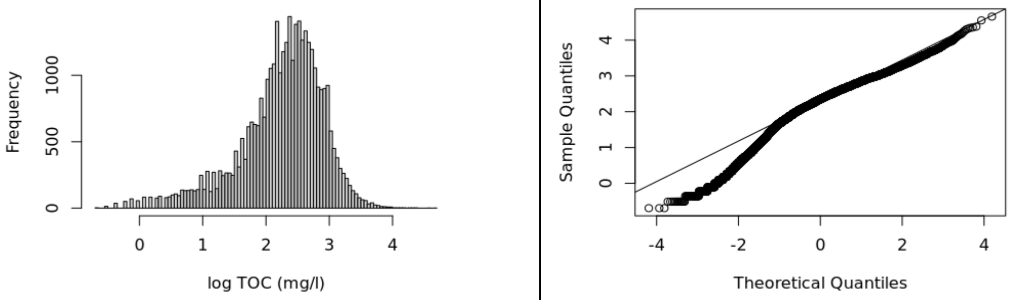
Variable	*Median*	*IQR*	*Min Median*	*Max Median*
year	2013	10	2001	2023
EC (mS/M)	4.7	5.4	1.28	56.56
sVISa	0.017	0.0071	0	0.03
Org. Charge Density (meq/g of C)	-6.7	2	-8.9	-4.02
TOC (mg/l)	10	7.8	1.15	26.6
Absorbance 420mm	0.17	0.19	0.01	0.53
ADOM/DOC	2	0.55	1.4	2.95
Field pH	6.8	0.75	4.47	7.94
Al3+ (µmol/l)	4.2	6.8	0.24	20.67
Si (mmol/l)	0.1	0.075	0.02	0.22
Cl (mmol/l)	0.079	0.2	0.01	0.82
Al total (µmol/l)	4.8	7.2	0.3	27
F (µmol/l)	5.3	5.3	0	28.68
NO3 (mmol/l)	0.0043	0.013	0	0.37
SO4 (mmol/l)	0.035	0.054	0.01	0.5
Alk Acid (mmol/l)	0.16	0.24	-0.07	3.93
Organic charge (meq/l)	-0.071	0.056	-0.2	-0.01
Sum of Cations (mmol/l)	0.44	0.49	0.12	5.76


(a) Histogram of sVISa

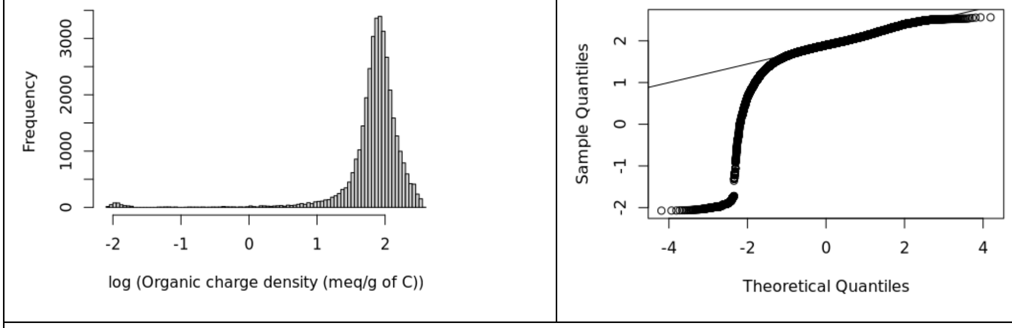
(b) QQ-plot sVISa


(c) Histogram of TOC

(d) QQ-plot TOC

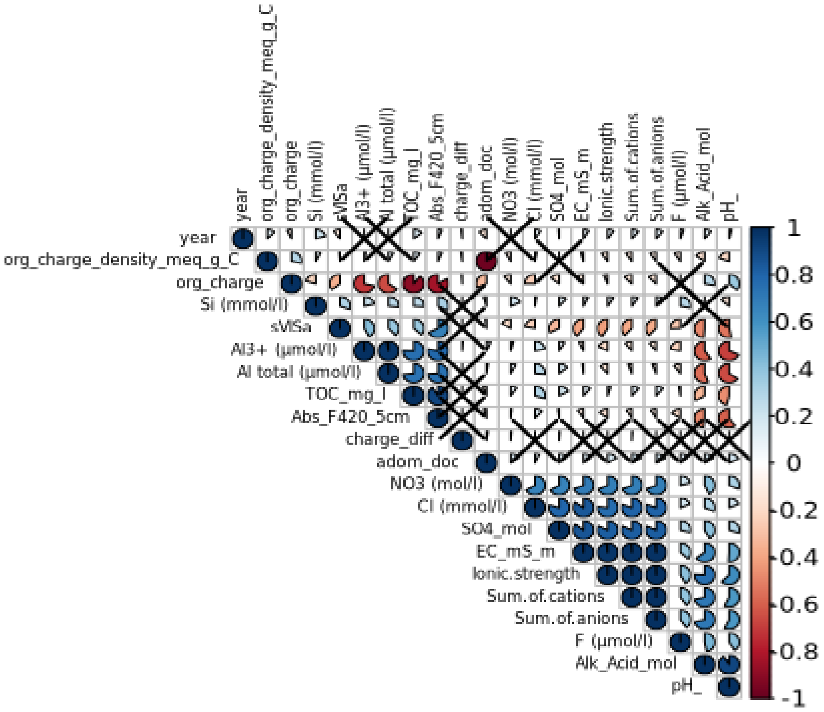

(e) Histogram of log(TOC)

(f) QQ-plot log(TOC)


(g) Histogram of log(OMCD)

(h) QQ-plot log(OMCD)

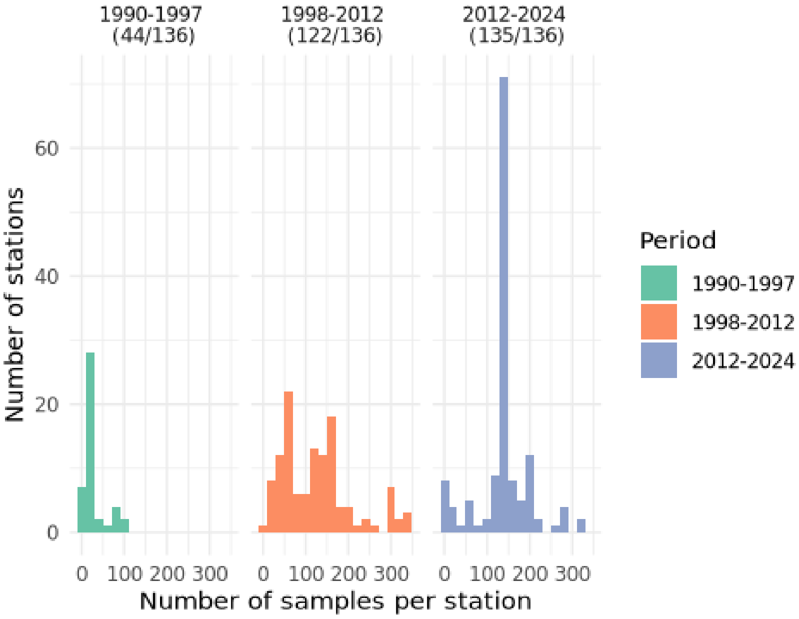
Appendix Figure A10: Evaluation of normality of sVISa (a&b), TOC (c&d), log(TOC) (e&f), log(OMCD) (g&f).



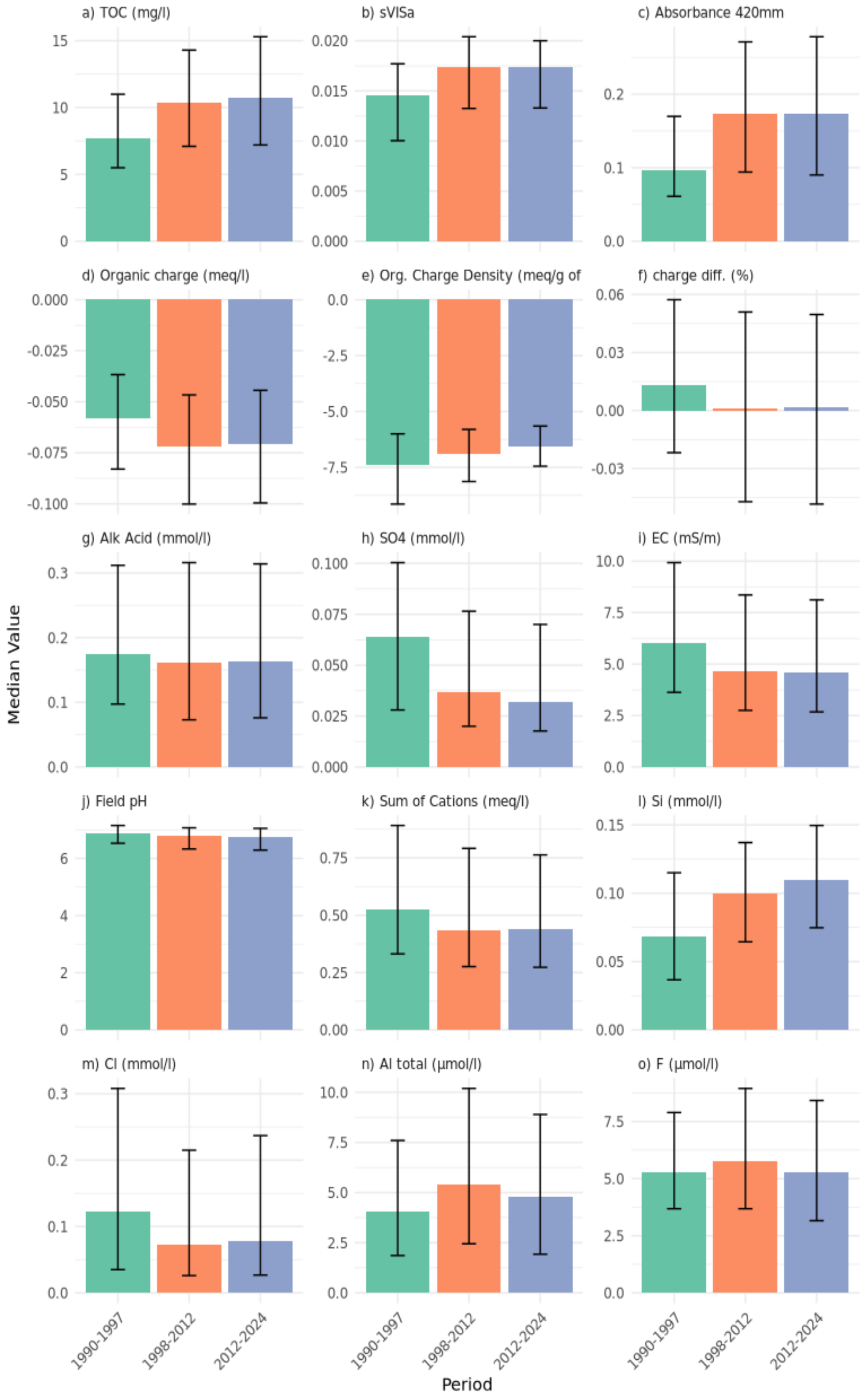
Appendix Figure A11: Correlation matrix using spearman (non-parametric) correlation to calculate correlation and significance of $p < 0.05$. Crossed out field indicate no significant correlation with $p > 0.05$. The area and color of the circle indicate the correlation coefficient.

Table A4: Water chemistry values measured and modelled for the stations used in this study. Data from all stations is weighted to be proportional to the number of samples in each station, such that each station has the same weight.

	1990-1997		**1998-2012**		**2012-2024**	
Variable	Median	IQR	Median	IQR	Median	IQR
EC (mS/M)	6	6.3	4.6	5.6	4.6	5.6
sVISa	0.015	0.0077	0.017	0.0072	0.017	0.0067
OMCD$_{5.6}$ (meq/g of C)	-7.4	3.1	-6.9	2.3	-6.6	1.8
TOC (mg/l)	7.7	5.5	10	7.2	11	8.1
Absorbance 420mm	0.096	0.11	0.17	0.18	0.17	0.19
ADOM/DOC	2.2	1.1	2	0.7	1.9	0.5
Field pH	6.9	0.62	6.8	0.74	6.8	0.76
Al3+ (µmol/l)	3.5	4.8	4.4	7.1	4.1	6.8
Si (mmol/l)	0.069	0.078	0.1	0.073	0.11	0.075
Cl (mmol/l)	0.12	0.27	0.073	0.19	0.079	0.21
Al total (µmol/l)	4	5.7	5.6	7.7	4.8	7
F (µmol/l)	5.3	4.2	5.8	5.3	5.3	5.3
NO3 (mmol/l)	0.0059	0.017	0.0045	0.014	0.0041	0.012
NO weak acid (%)	37	32	41	35	42	36
Strong acid (%)	49	35	38	29	37	30
Organic acid (%)	9.5	9.1	14	19	14	19
SO$_4$ (mmol/l)	0.064	0.072	0.036	0.056	0.032	0.052
Alk Acid (mmol/l)	0.17	0.21	0.16	0.24	0.16	0.24
Organic charge (meq/l)	-0.058	0.046	-0.072	0.053	-0.071	0.055
Sum of Cations (mmol/l)	0.53	0.56	0.43	0.52	0.44	0.49



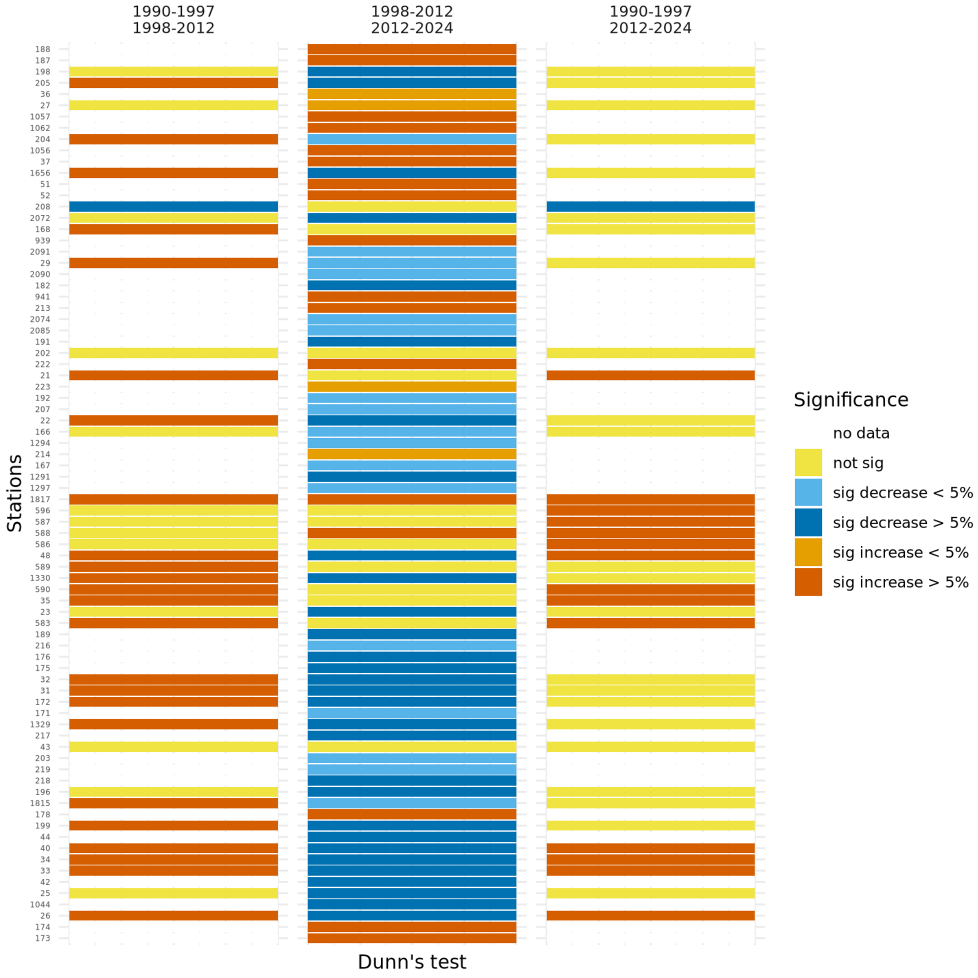
Appendix Figure A12: Histogram of the number of samples per station for each period. Stations with more than two samples were used in significance testing, the number of these stations are in the title as (number of station/total number of stations).



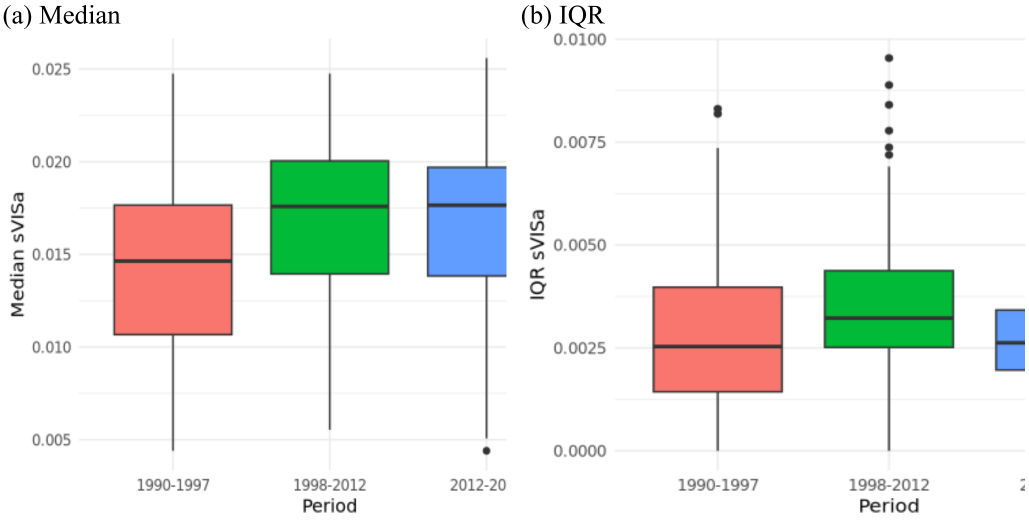
Appendix Figure A13: Bargraphs of water chemistry parameters modelled and observed for the three periods.

Appendix Table A4: Significance using Kruskal Wallis to determine stations which are significance for at least one of the comparisons (number of stations given in parenthesis) and then Dunn's test as a post hoc test to find the specific significant differences between the periods.

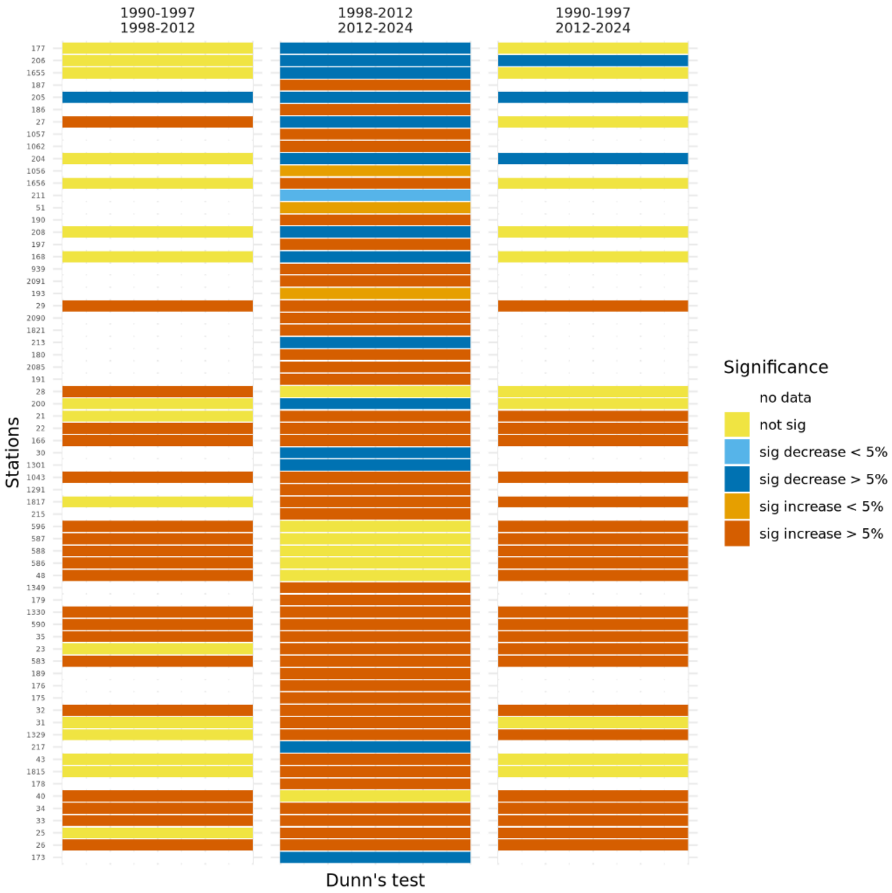
	period comparison	No data	Not Significant	Significant	Increase	Decrease
sVISa (80)	1990-1997 1998-2012	42	13	25	25	0
	1998-2012 2012-2024	0	12	68	47	21
	1990-1997 2012-2024	42	23	15	15	0
TOC (67)	1990-1997 1998-2012	30	16	21	20	1
	1998-2012 2012-2024	0	7	60	45	15
	1990-1997 2012-2024	30	11	26	23	3
$OMCD_{5.6}$ (77)	1990-1997 1998-2012	39	21	17	6	11
	1998-2012 2012-2024	0	8	69	60	9
	1990-1997 2012-2024	39	17	21	16	5
NA acid % (75)	1990-1997 1998-2012	39	14	29	29	0
	1998-2012 2012-2024	0	13	62	49	13
	1990-1997 2012-2024	39	6	37	37	0



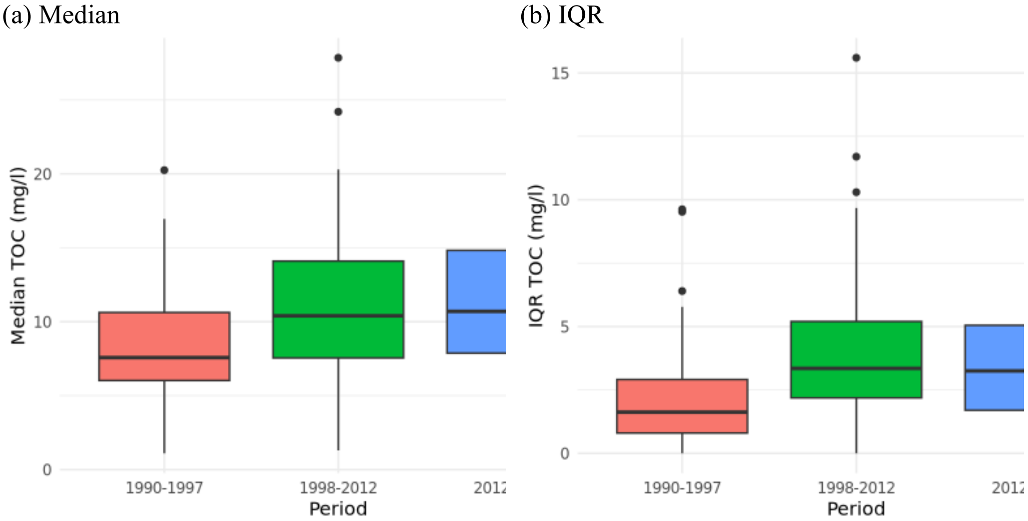
Appendix Figure A14: sVISa changes between periods. Only stations for which Kruskall-Wallis test found a significant difference between periods are included. In the tile is indicated how many station out of all the stations that had significant results between the periods had data for a comparison during the specified periods.



Appendix Figure A15: sVISa of all stations weighted.

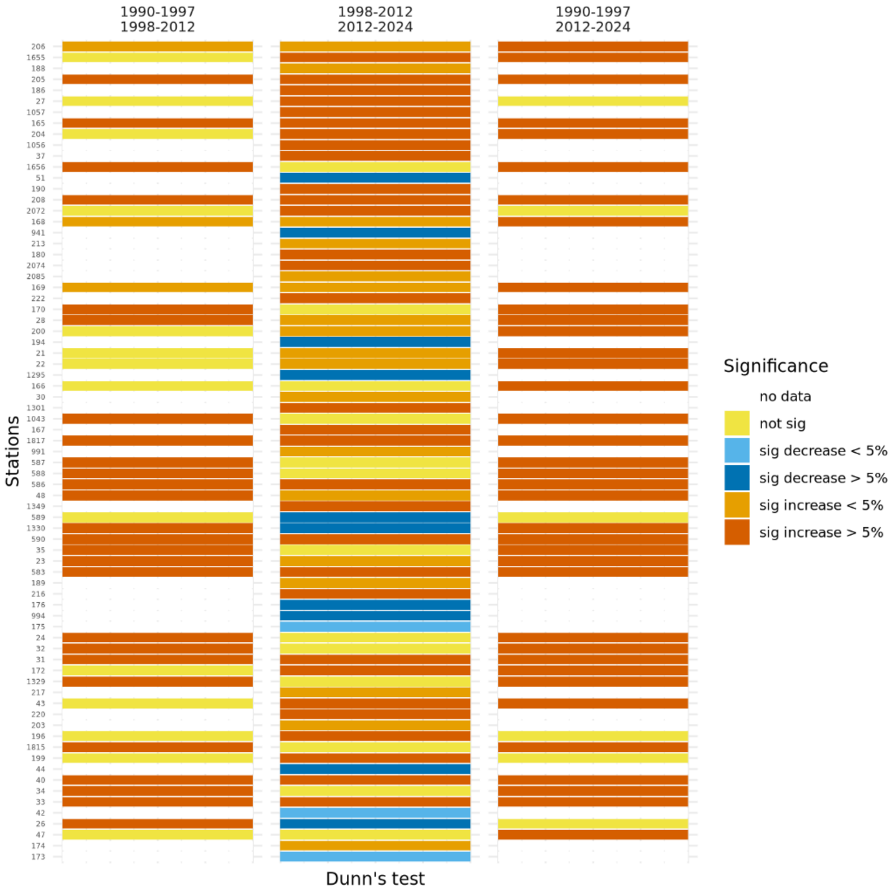


Appendix Figure A16: TOC changes between periods. Only stations for which Kruskall-Wallis test found a significant difference between periods are included. In the tile is indicated how many station out of all the stations that had significant results between the periods had data for a comparison during the specified periods.

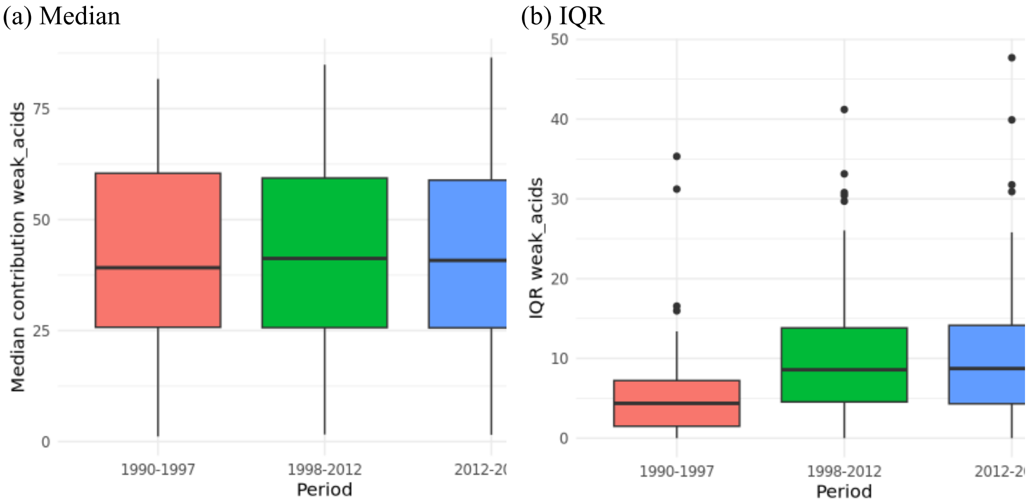

(a) Median

(b) IQR

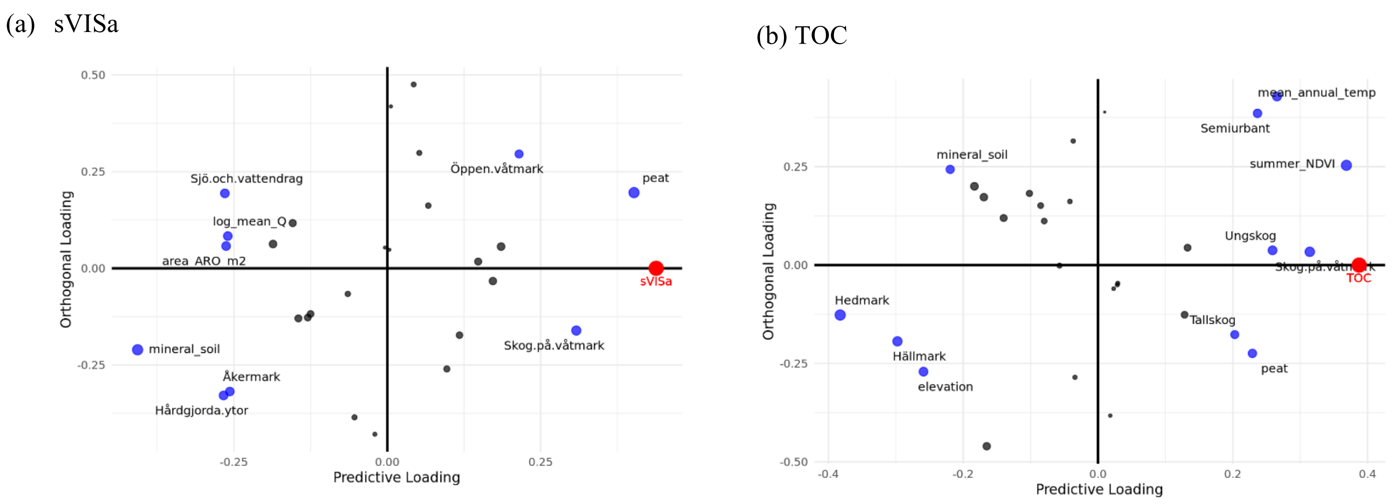
Appendix Figure A17: TOC of all stations weigthed.



Appendix Figure A18: Contribution of non-organic weak acids to charge changes between periods. Only stations for which Kruskall-Wallis test found a significant difference between periods are included. In the tile is indicated how many station out of all the stations that had significant results between the periods had data for a comparison during the specified periods.



Appendix Figure A19: Contribution of strong acids to charge of all stations not weighted.



(a) sVISa

(b) TOC

Appendix Figure A20: OPLS loading plot for sVISa (a) and TOC (b). Blue variables indicate a VIP score > 1 while black indicate a VIP < 1. The red is the response variable: sVISa. The x axis is the predictive axis indicating covariation with the response while the orthogonal axis is variation not explaining any variation in the response variable.

Appendix Table A6: OPLS results, showing the variation explained by the model (R^2) the model efficiency based on seven fold validation (Q^2) and the explanatory variables with a VIP score > 0.9.

$OMCD_{5.6}$		**TOC**		**sVISa**	
Variable	VIP	Variable	VIP	Variable	VIP
R^2	0.45	R^2	0.62	R^2	0.65
Q^2	0.20	Q^2	0.56	Q^2	0.59
samples	136	samples	136	samples	133
variables	30	variables	29	variables	29
area	-2.08	Hedmark	-2.01	mineral_soil	-2.14
Sjö.och.vattendrag	-1.81	summer_NDVI	+1.94	peat	+2.11
p75_Q_spec	+1.62	Skog.på.våtmark	+1.65	Skog.på.våtmark	+1.62
mean_Q_spec	+1.51	Hällmark	-1.56	Hårdgjorda.ytor	-1.40
log_mean_Q	-1.48	mean_annual_temp	+1.40	Sjö.och.vattendrag	-1.39
p25_Q_spec	+1.44	Ungskog	+1.36	area	-1.38
Åkermark	+1.38	elevation	-1.36	log_mean_Q	-1.37
Ingen.täckning	-1.33	Semiurbant	+1.24	Åkermark	-1.35
precipitation	+1.14	peat	+1.20	Öppen.våtmark	+1.13
Hedmark	+1.13	mineral_soil	-1.15	Hedmark	-0.98
Ungskog	-1.04	Tallskog	+1.07	Tallskog	+0.98
elevation	+1.01	area	-0.96	Ungskog	+0.91