EnISSA
enhanced in-situ soil analysis

EnISSA:
Enhanced In Situ Soil Analysis

NICOLE Technology Award 2010

Innovative site characterisation

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Summary

To meet the growing need for accurate and sensitive contaminated site investigation of large plumes of (semi) volatiles, a fast semi-quantitative in situ soil analysis method with low detection limits (ug/l-level) using the Membrane Interface Probe (MIP) is developed.

Accurate soil investigation and its sampling program is a critical component of reliable, efficient and cost-effective cleanup processes. Traditional sampling methods are characterised by low detection levels and a broad analysis spectrum. However delineation of the contaminated area by traditional sampling methods is not only a time and cost consuming task but also parts of the contaminated area could be overlooked. “On site” soil screening technologies such as the MIP and ROST techniques are already frequently applied to obtain additional information which cannot be obtained by traditional sampling methods. These techniques give detailed soil profiles on the field making “on site” decisions possible. However, they suffer from relatively high detection limits and cannot measure individual components (sum detectors). The purpose of the EnISSA technique is to combine the best of both worlds by generating on the field detailed soil profiles with low detection levels and a broad analysis spectrum, hence supporting reliable dynamic sampling plans.

Although MIP has proven its use in source zones survey, the characterisation of plumes is hampered by the elevated detection limits of the conventional detectors (PID, ECD, FID) compared to detection limits in off-site laboratories. Secondly, since the conventional detectors are sum detectors the information obtained by MIP cannot be compared with soil clean-up reference values which are determined for individual components. With the recent development of new detector types like the XSD for chlorinated volatile compounds, lower detection limits are possible, but they remain a-specific detectors. Therefore time inefficient and cost consuming extensive classic sampling methods are still needed and asked by regulators in a post-survey characterisation and confirmation phase.

The EnISSA method uses a GCMS system which is connected to the MIP by an innovative gas sampling system. A highly improved analysis method results in cycle times of 1 min. Using this technique, individual components can be characterised with detection limits in the range of 10 ug/l. The combination of smart method optimisation and innovative gas sampling is unique and makes it possible to qualify and quantify pollutant cocktails within the time frame of conventional MIP. Using the conventional MIP-speed (30 cm/min), each 30 cm a full characterisation of the pollution is possible on ug/l level using the proposed method.

Since individual components are measured well below the soil-cleanup values, both source and plume delineation is possible resulting in highly detailed soil profiles which allow reliable “on site” decisions. This makes it possible to delineate the entire source and especially the plume giving a well defined image of the contaminated area and reducing both sampling costs and time.

At this moment the EnISSA MIP has been validated and applied in demonstration projects. Further commercialization efforts are needed in order to promote the use of this new technology.

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1. Introduction

1.1 Problem statement

Accurate soil investigation and its sampling program is a critical component of reliable, efficient and cost-effective cleanup processes. Both the risk-assessment as the choice and dimensions of the remediation technique are based on the results of the soil investigation. Therefore the early stage of the sampling program will determine the final success of the remediation.

Overestimating the contaminated area can lead to an inefficient remediation design leading to higher costs then necessary. Underestimation of the contaminated area can lead to an incomplete remediation where the risks for the environment are not removed. Moreover, possible wrong BATNEEC evaluations can be made when the contaminated area is over- or underestimated.

Sampling strategies can be divided into traditional approaches and on-site screening technologies.

Traditional sampling methods are characterised by low detection levels and a broad analysis spectrum. However delineation of the contaminated area by traditional sampling methods is not only a time and cost consuming task but also parts of the contaminated area could be overlooked.

“On site” soil characterisation technologies such as the Membrane Interface Probe (MIP) and the Rapid Optical Screening Tool (ROST) are already frequently applied to obtain additional information which cannot be obtained by traditional sampling methods. These techniques give detailed soil profiles on the field making “on site” decisions possible.

Although MIP has proven its use in source zones survey, the characterisation of plumes is hampered by the rather high detection limits of the conventional detectors (PID, ECD, FID) compared to detection limits in off-site laboratories. Remark that the detection limit is highly dependent on experience and QA/QC of the MIP provider. Dependent on the detector used and the provider, detection limits on 200-500 ug/l level are possible if no soil matrix effects occur. Secondly, since the conventional detectors are sum detectors the information obtained by MIP cannot be compared with soil clean-up reference values which are determined for individual components. Therefore time inefficient and cost consuming classic sampling methods are still needed and asked by regulators in a post-survey characterisation and confirmation phase.

1.2. State of the art: Membrane Interface Probe

The Membrane Interface Probe (MIP) is a screening tool with semi-quantitative capabilities to measure volatile organic contaminants (VOC’s) in the subsurface, developed by Geoprobe®. Using different push technologies, the MIP is pushed in to the subsurface. Depth of 40-50 m-bgl are possible, dependent on geology.

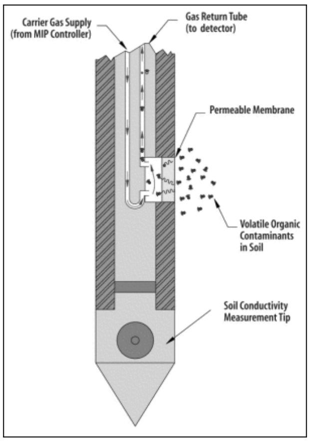


Fig. 1: Membrane Interface Probe (MIP)

The membrane is semi-permeable and is comprised of a thin film polymer impregnated into a stainless steel screen for support. The membrane is placed in a heated block attached to the probe. This block is heated to 120 C°. Heating the block helps accelerate diffusion of the contaminants through the membrane.

Diffusion occurs because of a concentration gradient between the contaminated soil and the clean carrier gas behind the membrane. A constant gas flow carries the contaminants to the gas phase detector at the surface.

The sensitivity and the detection limit of the MIP-method depend on which detector is used.

The most used detectors are:

- ***"Dry Electrolytic Conductivity Detector" (DELCD):*** Oxidation of chlorinated compounds generates ClO_2, which is conductive at high temperatures. Measurement of the electrolytic conductivity gives information on the amount of chlorinated compounds.
- ***"Photo Ionisation Detector" (PID):*** The PID measures the electric current which is generated by the ionisation of hydrocarbons through UV-light.
- ***"Flame Ionisation Detector" (FID):*** The FID ionizes hydrocarbons by using a flame. Measurement of the electric current gives an indication of the concentration level.
- ***"Halogen specific detector"* (XSD)**: The XSD oxidizes the halogen compounds by pyrolysis. Through adsorption and reaction of the free chlorine atoms with a alkali-sensitized cathodic surface, the thermionic emission of free electrons and halogen ions increases. The total cathodic current is an estimation of the amount of chlorinated compounds.

For the detection of volatile organic compounds, typically a combination of these detectors is used. For example BTEX and some chlorinated compounds will be detected by the PID detector. The DELCD is only sensitive for chlorinated and bromated compounds, while the FID will measure (flammable) organic compounds.

Quantification with the conventional MIP is difficult due to the use of sum-detectors. Since the detectors have a different response for different compounds, one cannot correlate the detector signal with concentration since conventional MIP is non-discriminable. Therefore the conventional MIP system can only give a indication of the contaminant concentrations.

Although the Membrane Interface Probe (MIP) has frequently been used for the delineation of VOC contaminated area's in plume zones, recent studies[1] give detection limits up to 10 000 ug/l for PCE. For BTEX the detection limit is between 1000-5000 ug/l. However, the detection limit is highly depend on the operators experience. Secondly, a new summation detector, XSD, is capable to measure 200 ug/l levels if no soil matrix effects occur. The lower concentration levels and the soil matrix effects on the sum-detector respons is also mentioned in literature[2].

Generally, the detection limits are much higher compared to for example the cleanup or norm values which are typically risk based. Therefore, conventional MIP is only applicable in the higher concentration zones and not readily applicable for plume investigations. Moreover, for the plume zone, a survey using sampling wells is still needed. This means that for the plume zone, only limited spatial information is obtained.

2. EnISSA MIP technology

2.1 EnISSA MIP

The purpose of the development of the EnISSA technique is to combine the best of both worlds (traditional sampling & on-site technologies) by generating on the field detailed soil profiles with low detection levels and a broad analysis spectrum supporting reliable dynamic sampling plans. With the EnISSA technique we want to find the optimal balance between measurements with a high analytical certainty and screening measurements which generates high spatial information.

The EnISSA technology should be characterised by a:
- broad analysis spectrum
- low detection limits
- detailed soil profiles (spatial information)
- information on the field

[1] Van Keer I., Bronders J., Touchant K., Wilczek D., Application of the Membrane Interphase Probe: An evaluation, Consoil LeS C.1 Sensors, 2008
[2] R.J. Stuut, Reactie op het artikel "10 jaar toepassing van de MIP: een evaluatie", Bodem, 3, 2008

The EnISSA technology comprises a MIP coupled to a GCMS system. The advantages of using a GCMS as detector, are the low intrinsic detection limit of the detector and the capabilities to measure individual compounds. To increase sensitivity and selectivity of the conventional MIP, methods using GC configurations combined with sample loops, purge and trap methods, adsorption tubes … have previously been established. Some of the efforts succeeded in lowering the detection limits to the range of cleanup or reference values. However the cycle times between measurements were 15-30 min limiting the practical and economical suitability on the field. As a result these techniques were not applied for commercial site investigations. For example, in order to decrease the chance of "missing" the contamination, each 30 cm a measurement can be needed. Therefore using a conventional GCMS measurement, each 30 cm the operator should wait for at least 15 min. This means that only 8-9 m could be screened in one day, which is not economically practical.

Within the EnISSA technology, the GCMS system has been modified in order to allow very short analysis times. To achieve this, two major innovative modifications were necessary.

First of all, the valve system which allows sampling of the gas in the MIP trunkline has been adapted for fast measurements. The whole inlet system to take samples from the trunkline and set these on the GC-column was reviewed and optimised to allow fast measurements.

The second modification is related to the GCMS method. By using a combination of an innovative GC configuration and a smart method optimisation, we were able to develop analysis methods with a very short cycle time. The first method, *"characterisation method"*, has a cycle time of 3-5 min. This method allows accurate determination of 54 volatile compounds. The second method, *"target method"*, has a cycle time of 1 min and can determine up to 12 predefined ("target") compounds.

As stated before, these short cycle times are crucial for the practical implementation of a MIP-GCMS on the field.

The overall probing speed of the conventional MIP is typically 30 cm/min. This speed incorporates a 30 sec wait-time to make sure that the probe is in equilibrium with the soil. During the next 30 sec, the probe is pushed 30 cm deeper. The "target method", with an analysis time of 1 min, fits into this time scheme and makes it possible to measure each 30 cm. This means that using the "target method" the probing speed is still limiting and not the analysis time.

2.2 Laboratory Results

Calibration of the EnISSA MIP was performed in the laboratory using standard solutions of PCE, TCE, benzene, toluene and xylene. Fig. 2 gives the calibration curves for benzene and TCE.

The laboratory calibration data show that the intrinsic detection sensitivity of the EnISSA MIP is about 10 ug/l for the BTEX and VOCl compounds. The linearity of the system is rather well. Also the response sensitivity, which can be defined by the ratio of the peak area and the concentration is high enough to quantify the measurements.

However there is a clear difference between laboratory and field conditions. It should be mentioned that the MIP – probe remains a semi-quantitative instrument due to the effects of the soil matrix on the MIP performance. For example, the amount of compounds which can be extracted from the soil is dependent on the heating front of the soil around the probe. This is hard to control and can have it's impact on the measured concentration levels.

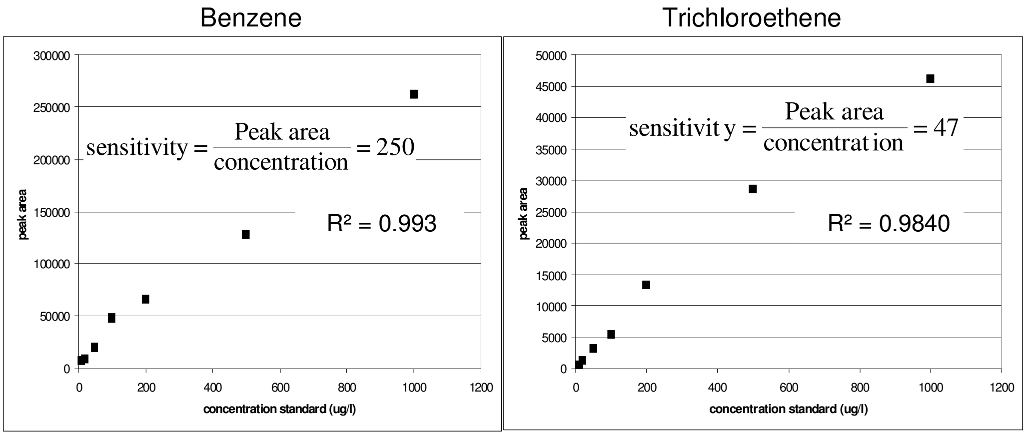


Fig. 2: Calibration data for benzene and TCE.

However, the field data which were obtained using the EnISSA MIP do not show that the soil matrix properties can change the order of magnitude of the result. This means that the EnISSA MIP is capable to support consistently decisions.

3. Case studies

Two case studies were selected to discuss here. The first one highlights the difficulties when trying to characterise a VOCl contamination using traditional sampling wells. It shows how the EnISSA MIP can contribute to a better CSM. The second case studies shows how degradation patterns of PCE can be visualised using the EnISSA MIP.

3.1 Tremelo (Belgium)

In 2007, a soil survey was performed on a site contaminated with PCE. Traditional MIP results indicated a potential contaminated zone starting at 6m-bgl. Sampling wells were placed with filter settings at 6-7 m-bgl and 10.5-12.5m-bgl. Groundwater samples at 6-7 m-bgl indicated the presence of PCE and TCE at concentrations of respectively 410 µg/l and 8.6 µg/l.

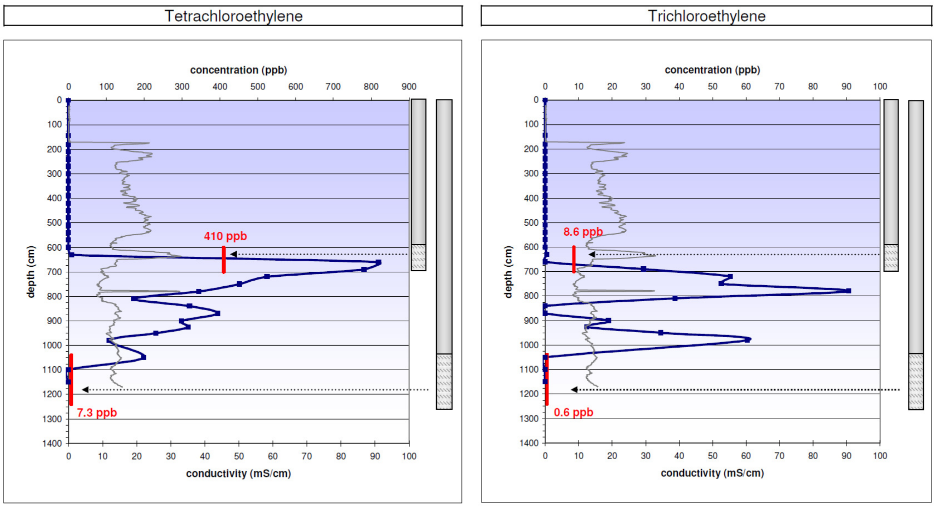


Fig. 3: EnISSA measurements for PCE and TCE compared to results obtained with a sampling well.

The EnISSA MIP was applied in the plume zone, near a sampling well. At 6.5m-bgl, PCE was found at concentrations up to 800µg/l. These high concentrations are located around a thin clay layer at 6.5 m-bgl. Taken into account the sampling volume using a traditional well, the EnISSA result matches very well with the lab results.

At 7.80 m-bgl a very thinl clay layer was found. Here, up to 90 µg/l of TCE was measured which is just above the cleanup reference value in Flanders (70 µg/l). As shown in the figures above, the EnISSA MIP profiles give a high resolution depth profile of the contaminations at ug/l level. It is clear that whereas the results of the two sampling wells underestimated the problem of TCE, the EnISSA MIP is capable of giving a high resolution soil depth profile for both PCE and TCE. Such soil depth profiles are of high value for defining efficient in-situ clean up strategies.

3.2 ‘s Hertogenbosch (The Netherlands)

A soil survey was performed on a former industrial site. A previous tradional soil sampling campaign indicated the presence of PCE, TCE, cis-DCE and VC. The purpose of the EnISSA MIP investigation was to identify the extent of the horizontal and vertical distribution of the contaminants. 16 EnISSA MIP’s were executed in both source and plume zone. After the EnISSA survey, sampling wells were placed based on the EnISSA results.

High concentrations were measured, but also the degradation products are clearly visualised by the EnISSA MIP (fig. 4). From the results, one can see that the less mobile compounds, PCE and TCE, occur in narrow regions in the soil. The more mobile compounds, cis-DCE and VC, are distributed over a broader region in the soil.

Such information could be of high value when assessing potential risks. For example the narrow region of PCE/TCE can easily be overlooked using a traditional survey based on sampling wells. Secondly, the information of how degradation is distributed in the soil, is very useful when selecting and designing a potential remediation strategy.

3.3 Other reference projects (non-limitative)

For more information on the following projects, please visit our website:

1. Machelen (Belgium): LNAPL characterisation: determination of not-suspected compounds
2. Waregem (Belgium): horizontal and vertical delineation of a PCE, TCE and DCE contaminated industrial site.
3. Oud-Heverlee (Belgium): Horizontal and vertical delineation of a PCE, TCE and DCE contaminated former dry-cleaner site.
4. Olst (The Netherlands): Delineation of an industrial site contaminated with naphthalene and BTEX.

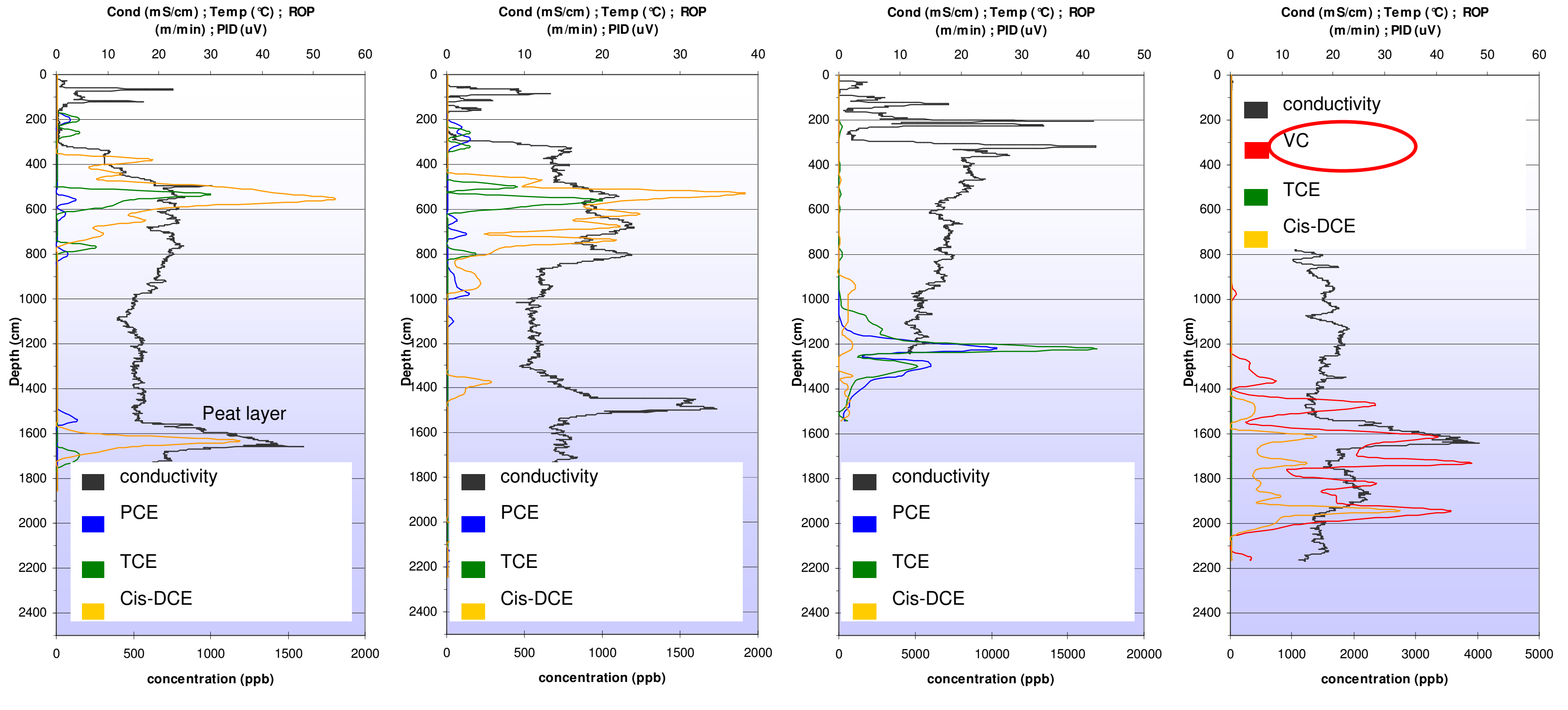
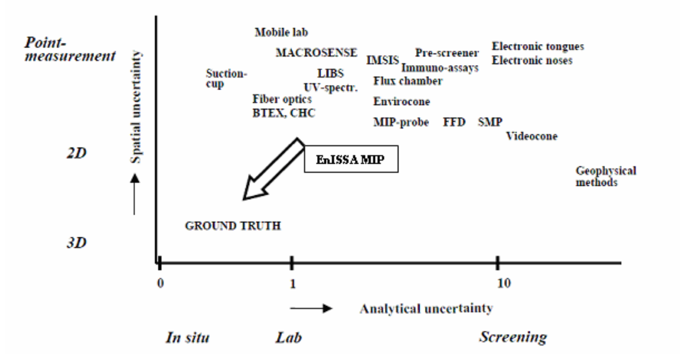


Fig. 4: Overview of EnISSA MIP logs

4. Impact on Conceptual Site Model (CSM)

The EnISSA method is characterised by a low spatial and a low analytical uncertainty. Therefore, using this technology within a Conceptual Site Model, the overall uncertainty can be drastically decreased (Fig. 5). Since the conventional MIP should only be used in the higher concentration area's while the EnISSA MIP can be applied in both source and plume zone, the spatial uncertainty for the EnISSA MIP is lower compared to the conventional MIP. Also since individual compounds are measured, the EnISSA MIP has a higher analytical certainty. Although the EnISSA MIP is still a semi-quantitative tool it generates data of high value which could be very important when setting up a conceptual site model.



Picture taken from Van Ree & Carlon, Land Contamination & Reclamation, 11 (1), 2003

Fig. 5: Combination of spatial and analytical uncertainty of different site characterisation technologies.

Since the EnISSA technology is capable to identify individual compounds in a semi-quantitative manner and can delineate both source and plume zone (Fig. 6), the EnISSA MIP makes it possible to visualize the whole contaminated area and obtain a three dimensional image of the individual compounds. This is not possible using the traditional method or the conventional MIP.

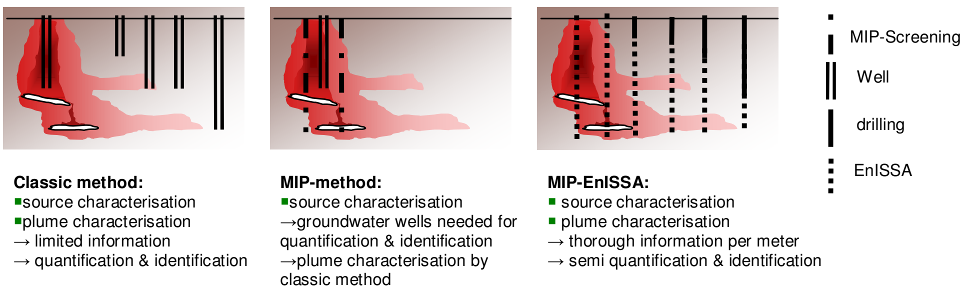


Fig. 6: Applicability of Classic sampling, MIP and EnISSA MIP.

Based on such a 3D image of the contaminated area, the strategic locations for confirmation wells can be determined. Limiting the number of sampling wells has of course a positive impact on time and costs.

Secondly the EnISSA MIP gives detailed vertical soil profiles for individual compounds. When the EnISSA MIP is applied in a horizontal screen, the possibility of "missing" a contaminated area is less, compared to sampling wells. Therefore, the EnISSA MIP assesses the risks, which can be caused by the contamination more accurately. For example DNAPL's or degradation pathways to more mobile and toxic compounds such as VC, which can occur in thin soil layers, can be characterised that can be overlooked easily using classic sampling wells.

In addition to a detailed characterisation of the contaminated area, the EnISSA results can be of high value for remediation design. Based on the high resolution depth profiles for individual contaminants, injections of for example carbon sources can be targeted. This leads to an improved remedial action resulting in a reduced consumption of reagents but without loss of performance due to a better balance between contaminant and, for example, injection reagents.

5. Cost savings

To estimate the cost savings which can be generated when using the EnISSA MIP in a site survey, the case study of 's Hertogenbosh, which was previously mentioned, is used. For this site a cost estimation for a traditional sampling approach was carried out and compared to the costs of the EnISSA campaign.

For this project, the EnISSA MIP was carried out at 16 locations in both the source and plume zone. To confirm the EnISSA results 6 sampling wells were placed near the EnISSA MIP's. Three sampling wells were placed with a filter setting between 15-20 m-bgl while the other sampling wells were above 15 m-bgl. Fig. 7 gives the costs for the entire project (EnISSA MIP + confirmation sampling wells).

These costs can be compared with the costs for a comparable traditional soil survey which covers the same area as with the EnISSA MIP measurements. To cover the interval which is measure by a MIP-log of 20 m-bgl, three sampling wells at 5, 15 and 20 m-bgl are necessary. For a MIP-log of 10 m-bgl, two sampling wells. Using these approximations, a total of 36 sampling wells is necessary to cover the same area as with the EnISSA campaign. A cost comparison between the traditional method and the EnISSA soil survey can be seen in Fig 7.

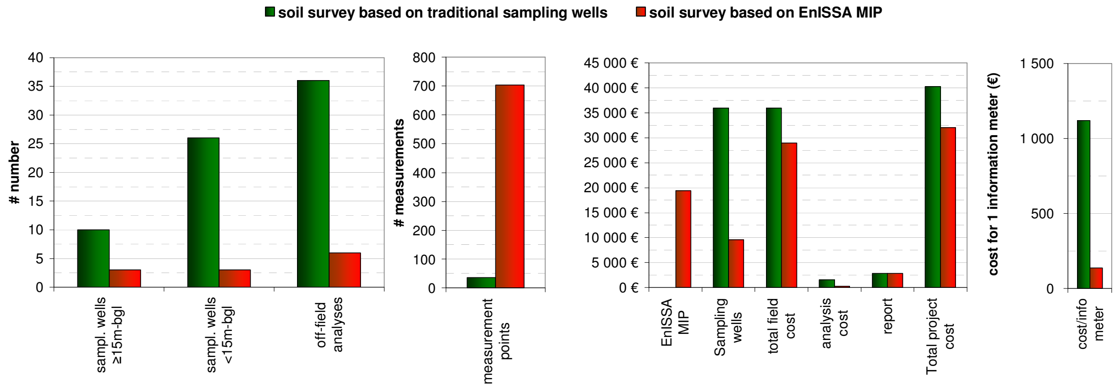


Fig. 7: Cost comparison between a traditional soil survey and a soil survey based on EnISSA MIP.

Fig 7. also shows the differences in number of sampling wells and off-site analysis. Even more important are the number of measurement points. These measurement points reflect the number of meters for which information is gathered. For the traditional method, only 36 points (sampling wells) are measured. Using the EnISSA method, up to 230 meters are investigated. Since each 30 cm a measurement is performed, a total of 700 measurement points was obtained.

Based on the cost comparison, it is clear that both the costs for field work and for off-site analyses are lower using the EnISSA approach. For the total project, the EnISSA soil survey costs about 8000 € less compared to the traditional soil survey. This means that a soil survey based on EnISSA can save about ¼ of the total project cost. Secondly, the cost per screened meter ("information meter") is much higher for the traditional survey (± 1100 €). For the survey based on the EnISSA MIP, the costs per information meter are only 130 €. Remark that the cost for placing confirmation sampling wells near some of the EnISSA MIP, are included in this cost per information meter.

The cost savings for the overall remediation project could be much higher since the costs which can be saved by using an optimised remediation design (ex. in-situ) based on the EnISSA results, are not implemented in this calculation.

6. Commercialization & prospects

6.1 Acceptance & Communication

Since conventional MIP is already accepted as a valuable screening tool by local authorities, we expect that the EnISSA MIP technology with a lower detection limit and more quantitative measurements will also be accepted for use in a site investigation. Therefore, the further validation and acceptance of the EnISSA MIP will be focused on the determination of reproducibility and accuracy compared with traditional sampling wells. This data will be available for authorities.

A first market communication was already done by several discussions with regulators, consultants, problem owners, contractors,… . The interest in the EnISSA MIP resulted in several demonstration projects in Belgium and The Netherlands. These projects were very successful and clearly demonstrated the strengths of the new technology.

The EnISSA MIP was presented on different international conferences such as Consoil 2010 and CSME 2010. These platforms are very important to reach a broad public. On these conferences, important contacts for possible future projects were made.

In addition to the presentations on conferences, we plan to participate at several workshops. A first demonstration workshop was already held in October 2010 by the VVMA (The Netherlands). This platform enabled us to demonstrate the technology to the consulting community in the Netherlands. Recently, we participated to a business trip to look for opportunities and contacts in Hungary. Upcoming events, such as the European project CityChlor (Nov 2010), the Ökoindustria Exhibition 2011 in Hungary, BEX in UK or Pollutec in France will also be important for further commercialization.

The consulting community, contractors and problem owners will further be contacted by sending an application brochure and invitations for demonstration projects.

Besides the attention on workshops and conferences, discussions with research institutes, such as VITO (Belgium), TNO (Netherlands), … can help with the further validation and acceptance of the technology.

Publishing in magazines such as BODEM and Probing Times will also contribute to the commercialisation. Possible publications in more scientific journals will be considered.

The EnISSA MIP technology was already offered in some public tenders from OVAM (Flanders), which are still in consideration. One of these tenders is related to the CityChlor project and is meant to evaluate different innovative characterisation technologies for chlorinated solvent contaminations. Such tenders could be very important for further commercialization and acceptance by the EU-authorities.

6.2 Market availability

The concern about soil contamination is becoming more and more important in almost all European countries. Certain regions have already taken legislative initiatives for quite some time. In Europe the majority of the members should still develop a legislation for soil protection and remediation. Therefore, there is a large growing potential for technologies which make it possible to assess the risks of soil contaminations and sustain the remediation plans.

Moreover, although authorities have taken legislative actions, the detailed characterisation of VOC contaminations with traditional approaches is often economically too expensive causing remediation plans to be postponed due to insufficient information on the distribution of the VOC contaminants in the soil. Therefore, innovative cost-effective technologies are needed in order to deliver information with is needed to make reliable decisions when selecting remediation strategies.

Due to the high costs of the remediation of VOC contaminations, remediation attempts which are enforced by several authorities impose in many cases a barrier on the continuity of the industrial sector. Often VOC contaminations occur at small and medium enterprises such as drycleaners or service stations. Therefore, recent soil remediation funds such as BOFAS and VLABOTEX in Flanders, but also BOSATEX in The Netherlands are raised, supporting these industries both financial and operational. These remediation funds start from a risk assessment which determines if remediation is needed. This risk assessment should incorporate the contaminants, DNAPL's, porosity, geotechnical data, … Important for these funds is a cost-effective remediation process. Therefore, a reliable method to characterize the contaminated site and measure soil parameters in order to assess the risks more accurately, could be of high value for these investigations, especially if this technique is cost-effective.

Since the EnISSA MIP generates detailed soil profiles on the field, it can be implemented in a dynamic sampling plan which results in an improved CSM and risk assessment. As a result, this technology could be of high value for the above mentioned remediation funds. Moreover the EnISSA MIP is a highly cost-effective technology resulting in cost savings for the overall remediation project.

6.3 Prospects

The EnISSA MIP has already been applied in some demonstration projects. We are planning to further contact potential clients by participating at congresses, workshops, exhibitions, forums, … . A website has been build, which will be updated frequently with reference project, further research results, … .

In addition to the communication to potential clients, we also still focus on further academic research to improve the EnISSA MIP system. A research project has been set up in cooperation with Group T (Leuven university college), focusing on different aspects of the calibration of the EnISSA MIP. One of these aspects is the application of the EnISSA MIP in different geologies which should increase our knowledge about possible effects of soil matrices on the measurements.

Secondly, a new design of a heated trunkline which transports the contaminants from the MIP probe to the GCMS will be developed. This highly innovative design should be able to heat the trunkline up to 200-300 °C and should make it possible to measure less volatile compounds such as PAH's. For this innovative design, we currently have a patent pending.

7. Conclusion

The EnISSA MIP makes use of an innovative GCMS system which is adapted to connect with the Membrane Interface Probe (MIP). This connection generates a MIP application with much lower detection limits compared to conventional MIP. Even more, the EnISSA MIP makes it possible to measure individual compounds. This is a major advantage compared to other on site technologies, which often measure sum-parameters.

The main purpose of the EnISSA MIP is to improve the conceptual site model. Since the EnISSA MIP gives an optimal balance between a low spatial uncertainty and a low analytical uncertainty, the EnISSA MIP can contribute to decrease the overall uncertainty in a conceptual site model. The three dimensional image of the contaminated site which is obtained using the EnISSA MIP makes it possible to define strategic locations for sampling wells and makes strategies such as search and remediate possible. Targeted remediation leads to an improved remedial action with in the case of in-situ remediation reduced consumption of reagents.

A cost comparison based on a real site survey, showed a cost saving of ¼ of the total project cost. The cost per screened meter for a soil survey based on EnISSA (including confirmation wells) is about 1/8 of the cost per screened meter for a traditional soil survey.