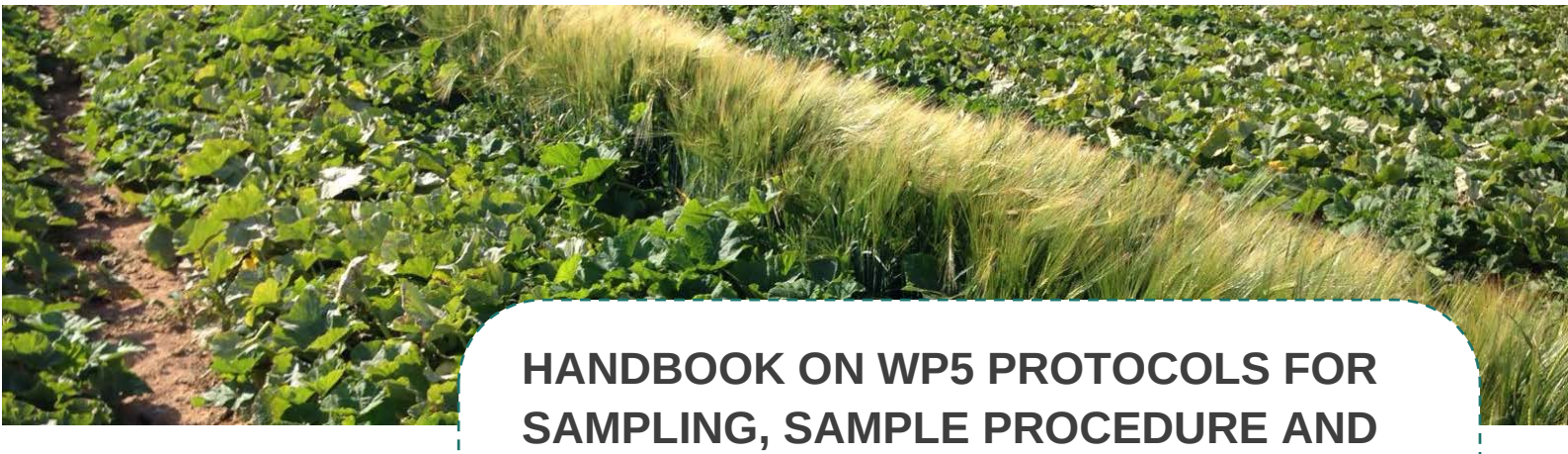
DIVERFARMING

Crop diversification and low-input farming across Europe: from practitioners' engagement and ecosystems services to increased revenues and value chain organisation



HANDBOOK ON WP5 PROTOCOLS FOR SAMPLING, SAMPLE PROCEDURE AND ANALYSIS

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E-mail of main editor	loczyd@gamma.ttk.pte.hu
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List of Diverfarming participants

Name	ACRONYM	COUNTRY
Universidad Politécnica de Cartagena (Coordinator)	UPCT	Spain
Consiglio per la Ricerca in Agricoltura e l'Analisi dell'Economia Agraria	CREA	Italy
Agencia Estatal Consejo Superior de Investigaciones Científicas	CSIC	Spain
Universita degli Studi della Tuscia	UTu	Italy
Asociación Regional de Empresas Agrícolas y Ganaderas de la Comunidad Autónoma de Murcia	ASAJ	Spain
Consorzio Casalasco del Pomodoro Società Agricola cooperativa	CCP	Italy
Arento Grupo Cooperativo Agroalimentario de Aragón	GA	Spain
Barilla G.E.R. Fratelli SPA	Bar	Italy
Disfrimur Logistica SL	DML	Spain
Universidad de Córdoba	UCO	Spain
Wageningen University	WU	Netherlands
Firma Nieuw Bromo van Tilburg	NBT	Netherlands
Industrias David S.L.U.	InDa	Spain
University of Portsmouth Higher Education Corporation	UPO	United Kingdom
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Weingut Dr. Frey	WDF	Germany
University of Exeter	Exeter	United Kingdom
Pecsi Tudomanyegyetem - University of Pecs	UP	Hungary
AKA Kft	AKA	Hungary
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Luonnonvarakeskus	Luke	Finland
Paavolan kotijuustola	PK	Finland
Polven juustola	PJ	Finland
Ekoboerderijdelingehof	EKO	Netherlands

Executive summary

With the long-term view of increasing diversification and biodiversity in Europe (CAP objective) and fostering sustainable development of bioeconomy, the Diverfarming consortium has come together to develop and deploy innovative farming and agribusiness models. Diverfarming will increase the long-term resilience, sustainability and economic revenues of agriculture across the EU by assessing the real benefits and minimising the limitations, barriers and drawbacks of diversified cropping systems using low-input agricultural practices that are tailor-made to fit the unique characteristics of six EU pedoclimatic regions (Mediterranean South and North, Atlantic Central, Continental, Pannonian and Boreal) and by adapting and optimising the downstream value chains organization through executing 14 field case studies and 8 additional long-term experimental plots.

Within Diverfarming, WP5 aims to provide sound and robust scientific understanding of the benefits and drawbacks of the tailored diversified cropping systems for improvement of the environmental quality and delivery of ecosystem services in each pedoclimatic region. In particular, this WP5 aims to evaluate the ability of tailor-made diversified cropping systems to: improve soil fertility and reduce soil and water contamination; improve soil structure and soil quality and increase soil C sequestration, foster natural soil functioning and promote the formation of stable aggregates; enhance C sequestration by biomass in perennial crops; reduce soil erosion rates; and reduce GHG emissions.

For this purpose, the first step is to develop a handbook to provide standardization of protocols among partners for field and laboratory investigations, so that by applying the same methodology we can effectively compare the results.

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1. Profile location

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

Geographical latitude loosely correlates with the position in bioms (physico-geographical zones), longitude often gives an idea about the distance from the ocean, and altitude informs about the location in lowland, hill, mountain or plateau.

Principle

The 3-D geographical locations of soil profiles must be unambiguously defined by geographical coordinates: latitude, longitude and elevation above sea level. The grid reference number (Universal Transverse Mercator, UTM) can be read directly from the topographic map. The latitude and longitude of the site should be given as accurately as possible (in degrees, minutes, seconds and decimal seconds) (FAO, 2006).

At present, the only acceptable method for the precise allocation of soil profiles is the application of a global navigation satellite system (GNSS). The commonly-used GNSS is the *Global Positioning System (GPS)* (Hofmann-Wellenhof et al., 1994), which is operated by the United States Department of Defense (DoD) and consists of a network of 24 NAVSTAR satellites orbiting the Earth on six different paths. Two complete orbits take just under 24 hours. A major benefit is that GPS works in all weather conditions.

The three main methods currently used for enhancing data accuracy are real-time differential correction, reprocessing real-time data and post-processing. To improve accuracy, GPS data are differentially corrected (Steede-Terry 2000). *Differential GPS (DGPS)* is based on the assumption that any two receivers placed relatively close to each other will experience similar atmospheric errors. DGPS requires a GPS receiver (base or reference station) to be set up at a precisely known location. Using an atomic clock, timing stability is ensured within one-millionth of a second. Integrating Doppler-derived speed with time signal reliability, an extraordinarily accurate distance measurement is achieved. The difference between the base station and the rover receiver is applied in real time in the field (Fig. 2.1.1.1).

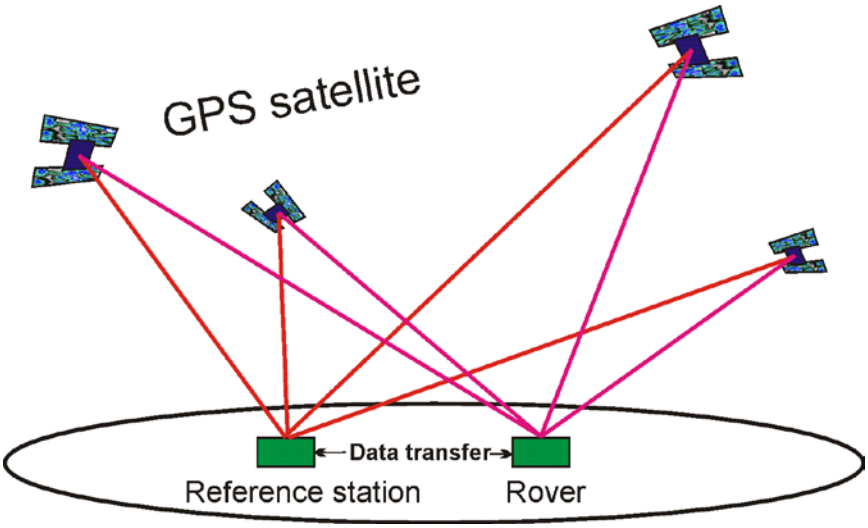

Figure 2.1.1.1 Differential global positioning

With *real-time DGPS* the base station calculates and broadcasts corrections for each satellite. The correction is received by the rover via a radio signal if the source is land based.

Some GPS devices have a built-in *altimeter*, which can give quite accurate (to within 3 m) readings of altitude above sea level.

Reagents

- None

Equipment

- Topographic map at 1:25,000 or 1:10,000 scale.
- The most widely used Trimble GPS devices:
- Juno 5 Series – The Juno 5 Series offers smartphone-like operation and compatibility with Trimble mapping and GIS software.
- Geo 7 Series – Equipped with cutting-edge Trimble Flightwave remote positioning technology.
- Yuma 2 – a solidly constructed tablet specifically designed for field applications.
- TDC100 – works like a smartphone, but with enhanced GNSS capabilities.

Procedure

a. Check settings for coordinate system (NRC 1993; EPA 2015),
b. Set accuracy threshold (required accuracy),
c. Read and save latitude and longitude coordinates in dd.dddddd format.
d. Convert coordinates into degrees/minutes/seconds format.

Remarks

- Now a series of modern smartphones (including Huawei Honor 7X Smartphone Android 7.0, Huawei Honor 9 Lite Smartphone Android 8.0, Huawei Honor V10 Smartphone Android 8.0, Samsung Galaxy Mega 5.8 GT-I9152, Samsung Galaxy S6 SM-G920V, Samsung Galaxy S7 G930V, Samsung Galaxy S7 EDGE G935V, Sony Ericsson Xperia Z3 Compact, Sony Xperia Z5 Compact E5823 etc.) are also capable of site positioning with satisfactory accuracy.
- The Galileo system, now under development, will be operated by the European Global Navigation Satellite Systems Agency (GSA) of the European Union. It will be a new alternative global navigation satellite system, which will remain under civilian control. In the near future, further satellites will be launched to enlarge the constellation, gradually improving Galileo availability worldwide. Its full operational capacity (FOC) of 30 satellites (24 satellites plus six orbital spares) is expected to be accomplished by 2020.

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2. Major landform

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

The relief of hilly terrains influences soil distribution, water availability and crop growth in a complex way (FAO, 2006). The landform units identified are used for site description in soil and vegetation mapping and in landscape ecology (landscape pattern). For sustainable agriculture, it is important that the typical geometry of the landform where the plot is situated influences land management opportunities and erosion hazard. Particularly, the DEM-based delineation and classification of landforms can assist in land use optimisation and farming practices design (Seif 2014).

Principle

1. The dominant criteria of major landforms are general slope and relative relief (relief intensity) (FAO, 2006). The relief intensity (expressed in m km^{-1}) is the difference between the highest and lowest point within the terrain unit per distance specified for the actual purpose of study (Table 2.1.2.1).

Table 2.1.2.1 Identification of major landforms from topographic parameters (FAO, 2006)

1st level	2nd level	Gradient	Relief intensity	Potential drainage density
		(%)	(m km^{-1})	
L level land	LP plain	< 10	< 50	0-25
	LL plateau	< 10	< 50	0-25
	LD depression	< 10	< 50	16-25
	LV valley floor	< 10	< 50	6-15
S sloping land	SE medium-gradient escarpment zone	10-30	50-100	< 6
	SH medium-gradient hill	10-30	100-150	0-15
	SM medium-gradient mountain	15-30	150-300	0-15
	SP dissected plain	10-30	50-100	0-15
	SV medium-gradient valley	10-30	100-150	6-15

T steep land	TE high-gradient escarpment zone	> 30	150-300	< 6
	TH high-gradient hill	> 30	150-300	0-15
	TM high-gradient mountain	> 30	> 300	0-15
	TV high-gradient valley	> 30	> 150	6-15

2. In the simplest way, the geomorphological environment of the agricultural plot, i.e. the landform type on which the plot is located, can be described from a geomorphological map. Depending on the source of elevation data (resolution), geomorphological maps based on DEMs can be very accurate and allow classification observing subtle changes in elevation (Smith et al., 2011). The geomorphon approach is a grid method to identify landforms at different scales (Józsa & Fábián, 2016).

3. Recently, automated techniques of landform classification have been developed (Blaszczynski, 1997). Most methods are based on the *Topographic Position Index (TPI)*, which is an ArcView GIS application. It shows the difference in elevation between a given cell and the cells in its vicinity (Jenness, 2006):

$$TPI_i = M_0 - \sum_{n-1} M_n \, n^{-1} \quad [-] \qquad \text{(Eq. 2.1.2.1)}$$

where

M_0 is the elevation of the model point (cell) under evaluation,

M_n is the elevation of the grid cell,

n is the total number of surrounding points employed in the evaluation.

Landform classification can be made more accurate through the addition of further topographic metrics, such as elevation, slope gradient or exposure. Combined with slope gradient, the TPI index allows the differentiation of six classes: ridge, upper slope, middle slope, lower slope, flat slope and valley. Altering the diameter of the cell vicinity, Weiss (2001) obtained increased accuracy of landform classification. The landforms can be analysed statistically and correlated with agricultural land-use classes.

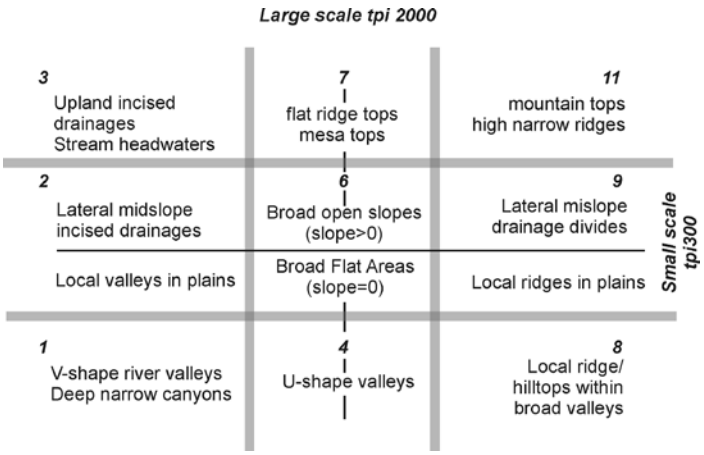

Figure 2.1.2.1 Combining TPI at two scales to identify landform classes, numbers are landform values (after Weiss, 2001)

A well-known algorithm of landform classification (Jenness, 2006) uses a multi-scale approach by fitting a quadratic polynomial to a given window size applying least squares. A product of the method is shown in Fig. 2.1.2.2. A great advantage of this approach is the fact that the definition of classification criteria can be flexibly modified by the user.

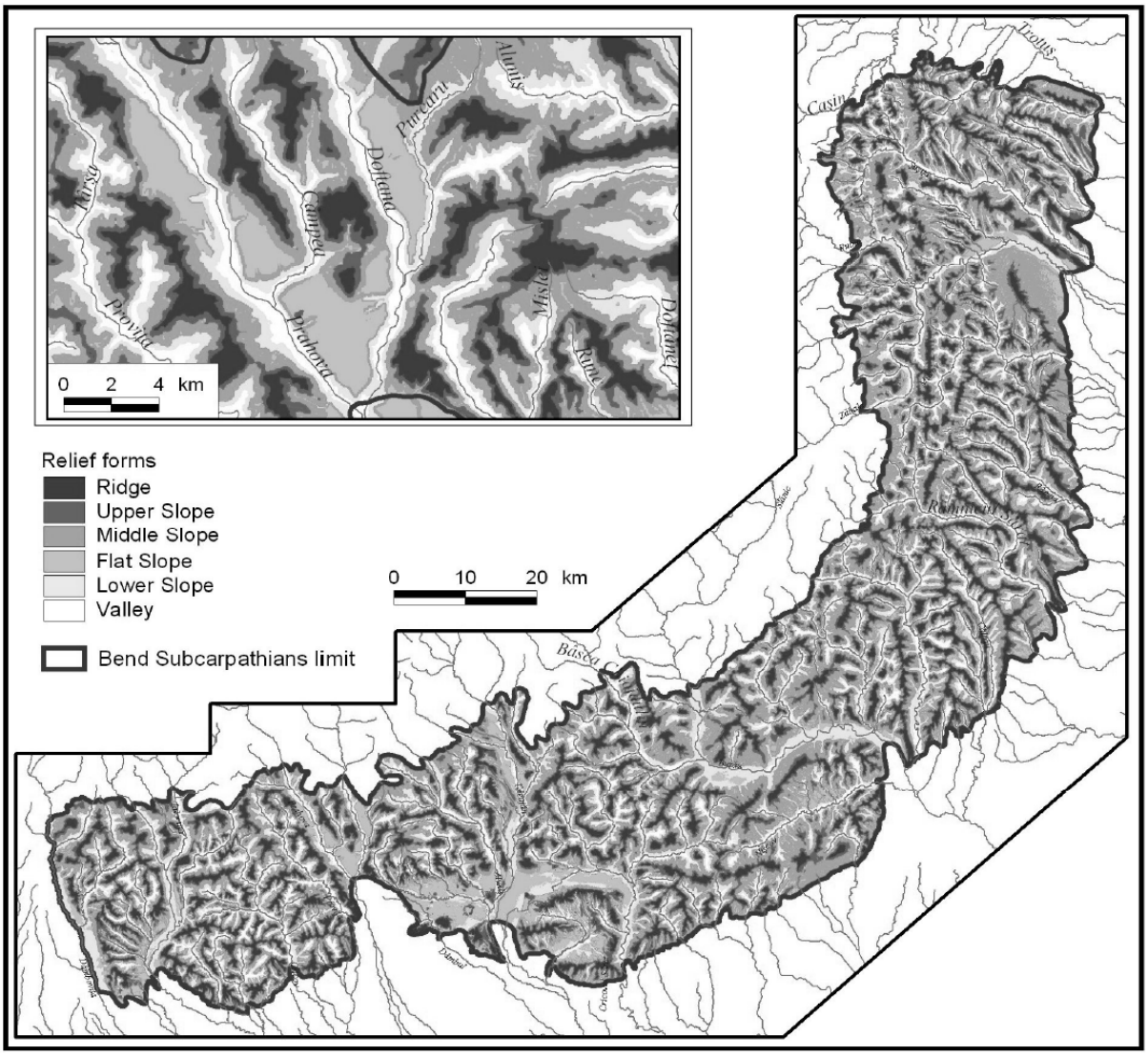

Figure 2.1.2.2 Landform types in the Curvature Carpathians, Romania, generated from Digital Elevation Model using the TPI index (after Chendeş et al., 2009)

4. *Geometric fuzzy land element classification* (Schmidt & Hewitt, 2004) is also based on the fundamental properties of land elements, i.e. local geometry and scale. Since there is a high degree of uncertainty in their delineation and semantic descriptions, land elements have to be fuzzified. An advantage of the model is that it requires a relatively limited number of parameters. 'Object-based image analysis' tools have the ability to segment and classify DEMs into representative objects arranged in a multi-level hierarchy. Ambiguities in landforms both in attribute and geographical space are properly reflected in the fuzzy classification (Gerçek et al., 2011). The methodology for modelling land elements is implemented as a two-step process (Schmidt & Hewitt, 2004): first, form elements are classified based on local geometry, and second, land elements are derived by evaluating the form elements in their landscape context.

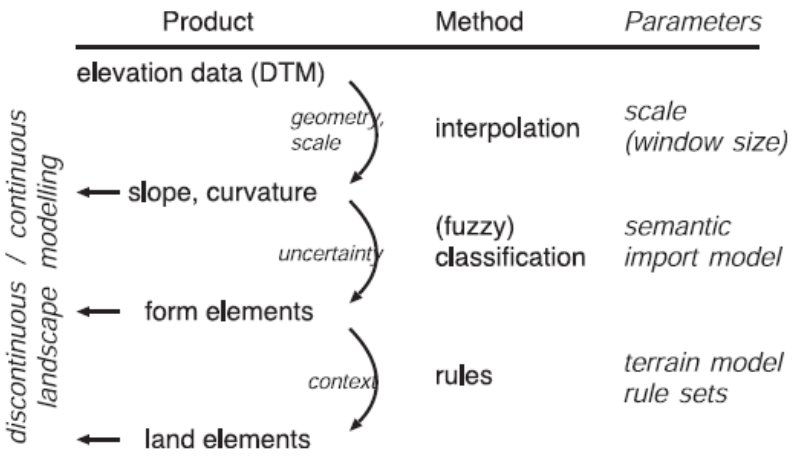

Figure 2.1.2.3 The principles of the fuzzy delineation of landscape elements (after Schmidt & Hewitt, 2004)

5. Remote sensing approaches. The application of radar inferometry for landform classification (Widyatmanti et al., 2016) is based on the *combination of* multispectral imaging, *DEM and radar interferometry* (Fig. 2.1.2.4). The classes obtained through DEM segmentation using InSAR Imagery (MacMillan & Shary 2009) are volcanic, structural, fluvial and karst landforms differentiated by elevation, slope gradient, relief dissection and curvature ("toposhape" features). An advantage of this classification is that it also provides genetic information from morphometric parameters.

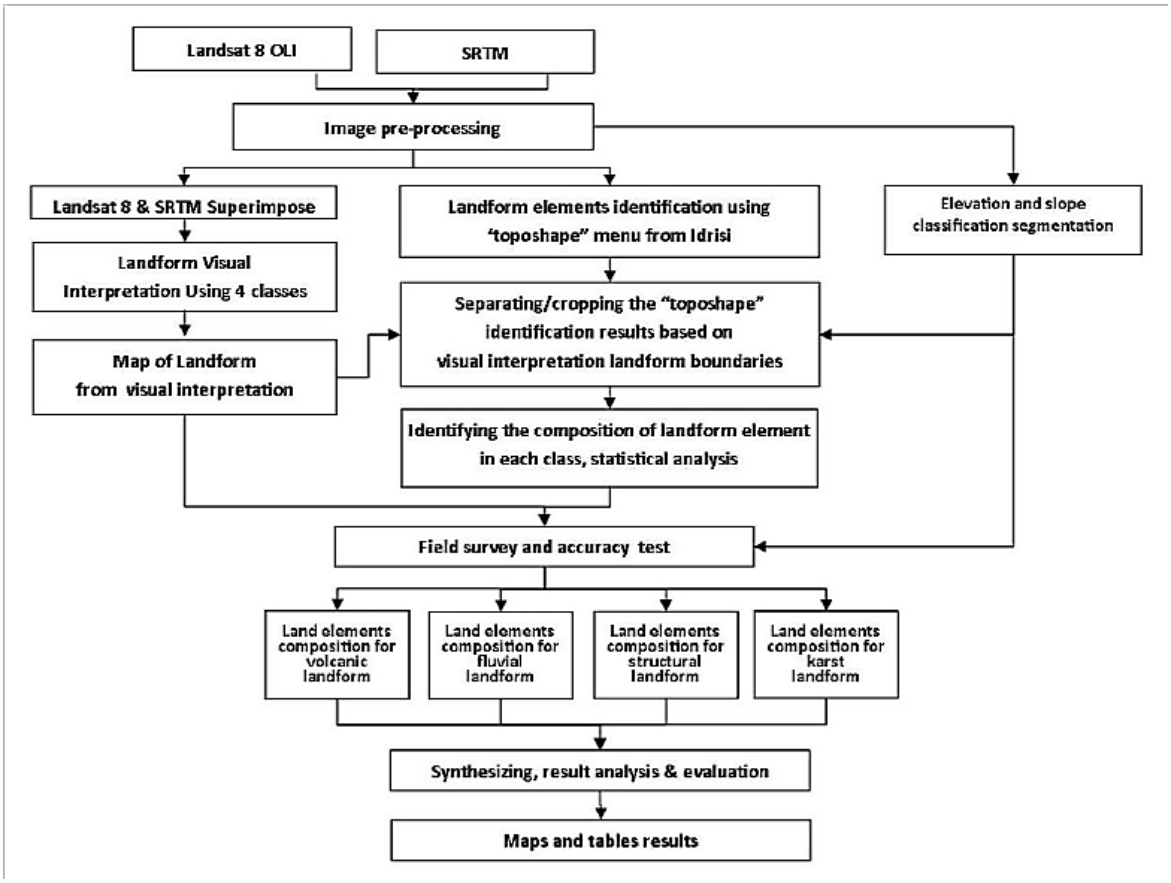

Figure 2.1.2.4 Flow chart of land elements classification (Widyatmanti et al., 2016)

Procedure

1. Classification from topographic map (FAO method):

1.a. Classify terrain as level, sloping or steep land visually from a topographic map (1st level) (FAO, 2006)

1.b. Distinguish classes based on relief intensity and potential drainage density (2nd level)

2. Classification by parameters derived from DEM

2.a. Define the lookup distance (maximum scale of mapping), the topographic grain, establish the characteristic local ridgeline-to-channel spacing (Pike et al., 1989)

2.b. Manually find a break-point where the increase rate of local relief is significantly reducing

2.c. Use a supervised classification algorithm to identify and map physiographic units at the appropriate scale (Józsa & Fábián, 2016)

3. Automated classification based on TPI:

3.a. Add the elevation or surface model grid to Spatial Analyst for ArcView 3.x software (Jenness, 2006)

3.b. Select neighbourhood type and radius

3.c. Generate Slope Position Classification

3.d. Select the themes and the classification criteria in the Slope Position Analysis dialogue

3.e. Select and define a classification regime

3.f. Load criteria sets

3.g. Confirm your selected classification criteria in the Landform Analysis dialogue

3. Fuzzy land element classification:

4.a. Create a generalised terrain and parameterise it by scaled derivatives (slope gradient and curvature). These scale-dependent derivatives parameters are calculated at varying window sizes (Wood, 1998)

4.b. Fuzzify local landform geometry. Slope gradient is continuous, and curvature is referred into three classes: concave, straight, and convex

4.c. Generate membership-value maps for each of the 15 form elements

4.d. Identify two moving window sizes for the horizontal spatial scales and specify an elevation threshold to model landforms in terrain context (Schmidt & Hewitt, 2004)

4.e. Reclassify landforms within the terrain context, using a set of rules; combine the higher scale landscape position model with the form element model

5. Classification using satellite imagery:

5.a. Interpret Landsat 8 imagery (multi-band composites) visually to draw the boundaries of landform types and to identify topographic elements

5.b. Use the multi-level landform mapping approach by Van Asselen and Seijmonsbergen (2006) to obtain more accurate landform interpretation

5.c. Define training sites in the study area for supervised classification

5.d. Segment the DEM into high and low elevation classes and by slope gradient and elevation (Table 2.1.2.2)

5.e. Analyse landforms by “toposhape” (using Idrisi package) and make the segmentation (using eCognition package) per unit area based on landform boundaries

5.f. Overlay drainage map and establish landform types relative to drainage lines (Saadat et al., 2008 – Table 2.1.2.3)

Table 2.1.2.2 DEM segmentation by elevation and slope gradient (Widyatmanti et al., 2016)

Class	Elevation – relative height (m)	Slope (%)
1	< 50 : lowlands	0-2 : flat or almost flat
2	50-200 : low hills	3-7 : gently sloping
3	200-500 : hills	8-13 : sloping
4	500-1000 : high hills	14-20 : moderately steep
5	> 1000 : mountains	21-55 : steep
6	-	56-140 : very steep
7	-	> 140 : extremely steep

Table 2.1.2.3 Landform classification scheme according to Dessaunettes et al., (1971)

Landform	Slope class	Elevation range	Specific characteristics	Land use
River Alluvial Plains (RP)	< 1%	< 150 m	a, These landforms are close to a river	Usually used for irrigated farming
			b, General slope direction is parallel to that of the river. The general shape of this landform is an elongated eclipse with the major axis parallel to the slope of the river	
			c, These landforms are usually next to a meandering river	
Piedmont Plains (PD)	0–5%	n.a.	a, Since RP is really a subset of PD, then RP must be isolated first	Usually used for irrigated or dryland farming
			b, The shape of PD is normally one of a trip with the long dimension parallel to the mountain range front. The transverse elevation cross-section of PDs is normally quite flat	
			c, PDs are restricted to areas with non- or slightly gravelly soils	
Gravelly Talus Fans (GFc)	0–5%	n.a.	a, GFc and GFr are as a result of a major water course or a number of smaller water courses running down to the foot of mountain range fronts	Rarely used for irrigated or dryland farmed

Gravelly River Fans (GFr)	Mostly 0–2%, occasionally 2–5% in the higher parts	n.a.	b, The shape of an individual GFc and GFr is usually triangular-shell shape. A group of GFc/GFr is normally one of a strip with the long dimension parallel to the mountain range front. The transverse elevation cross-section of these fans is clearly convex	
			c, GFc and GFr always have highly gravelly soils	
Plateaux and Upper Terraces (TR)	0–12%, with local relief intensity feature slopes of up to 25%	> 500 m		Tops usually used for dryland farming
Hills (H)	Mostly 8–25%	50–500 m		Usually grazing and/or forestry
Mountains (M)	Over 25%	Mostly 500–1500 m		Usually grazing and/or forestry
Lowlands (LL)	Usually < 1%	< 150 m	a, These landforms are located at the lowest elevations of watersheds	
			b, The transverse cross-section of LL is nearly level and often concave	
			c, The water-table level is usually above the ground surface. The ground and surface water tends to accumulate with subsequent accumulation of fine sediment and salts	
Floodplains (FP)	Usually < 1%	<150 m	a, These landforms are usually next to a river known to flood frequently	
			b, The transverse cross-section of FP is nearly level	
			c, FPs are affected by incoming surface water flow	

Remarks

- Although crop yield variability is largely explained by soil and terrain properties, most agricultural land-use types can also be characterised by landforms identified by a topographic index derived from DEM. This index is relevant in assessing the variability induced by topography on climatic conditions and crop management.
- Landform classification approaches are very similar to each other, but their products may differ greatly in scale. Therefore, multi-scale techniques are preferred.

- Unsupervised automated landform classifications reflect the frequency distributions of the input variables rather than pre-set criteria. The results must be analysed and calibrated empirically.

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3. Position in landscape

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

Position on the landform influences soil-water relationships, microclimate and vegetation. Straight slopes allow maximum slope length and slope wash can shape their surface unimpeded. In contrast, on complex slopes localised breakpoints appear and reduce slope length and, thus, the generation of sheet wash and concentrated forms of soil erosion (rills and gullies) (Sensoy & Kara, 2014).

Principle

According to the *catena principle*, soil types follow one another along slopes in a more or less predictable sequence (Gerrard, 1992). The automatic extraction of landforms through discretising a DEM begins with defining a simple succession of peak – slope – horizontal surface – depression. Further refinement of landform classification is possible by applying the *Topographic Position Index (TPI)* (Chendeş et al., 2009) (see Chapter 2.1.2 on Major landform) or the *nine-unit slope model* (Fig. 2.1.3.1 – Dalrymple et al., 1968) or vertical and horizontal slope curvature (Young, 1972).

Reagents

- None

Materials and equipment

- None

Procedure

1. The locations of study plots are defined on the 9-unit slope model (Fig. 2.1.3.1 – Dalrymple et al., 1968).

    1.a. Stratify by scale into landscapes, landforms and microfeatures
    1.b. Discretise slope by gradient classes
    1.c. Identify predominant geomorphic processes (fluvial, eolian, etc.)
    1.d. Distinguish between landform elements: interfluve, valley shoulder (seepage slope), convex upper slope, fall face, midslope, colluvial footslope, alluvial toeslope, channel bank, riverbed

2. Alternatively, vertical and horizontal slope curvature is used to establish the types of slopes where the plot is located (Fig. 2.1.3.2 – Young, 1972).

    2.a. Establish straight (S), convex (V) and concave (C) slopes in planform
    2.b. Establish the same types in lateral view
    2.c. Identify terraced and other complex (irregular) slope shapes

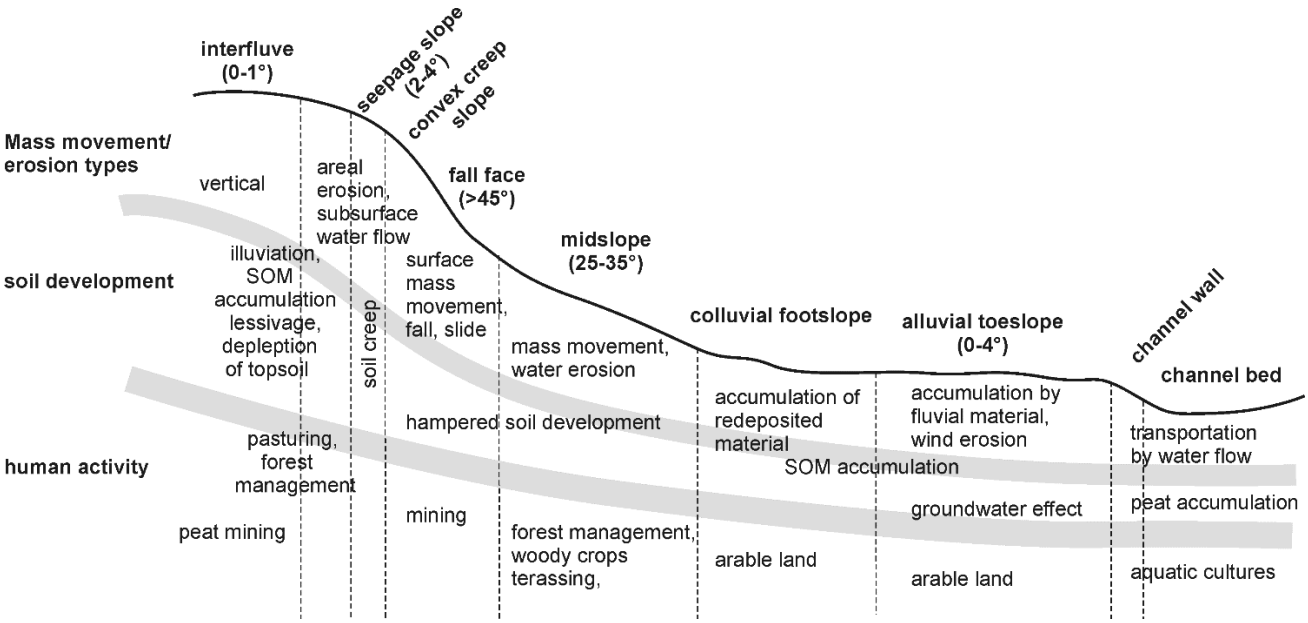

Figure 2.1.3.1 The nine-unit slope model (after Dalrymple et al., 1968), soil development and human activity

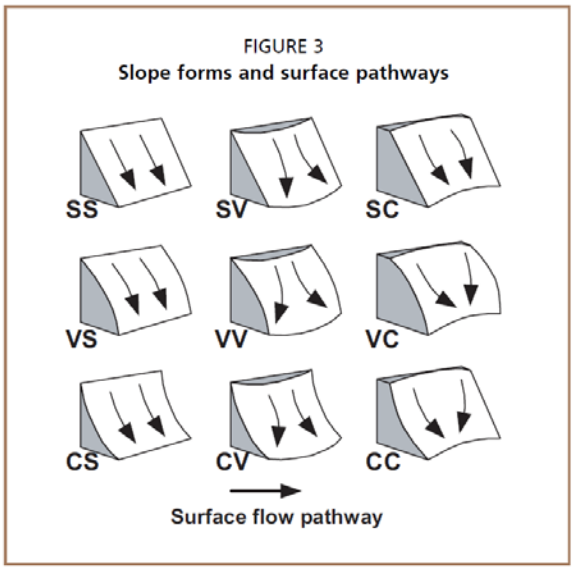

Figure 2.1.3.2 Slope curvature types influencing runoff

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4. Slope gradient

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

The topography of an agricultural field determines how susceptible the soil is to erosion by water. Among the topographic factors the slope gradient is the most important in governing the rate of soil erosion (Zingg, 1940; Assouline & Ben-Hur, 2006). In general, the steeper and longer the slopes are in a field, the greater the soil erosion potential (OMAFRA, 2016). The close relationship between slope gradient and soil erosion must be taken into account in planning land use in hilly areas (Marsh, 2014). The relationship between slope gradient and soil erosion, neverthless, varies considerably with different land use classes (Fig. 2.1.4.1) and land management practices (Liu et al., 1994).

Through soil erosion, slope gradient also controls the loss of nutrients from plots. For instance, a study in Poland (Chowaniak et al., 2016) found that annual average losses of calcium and magnesium were the highest from plots with gradients above 16% (on average, 25% higher for Ca and 26% higher for Mg than losses measured on plots with a 9% slope gradient).

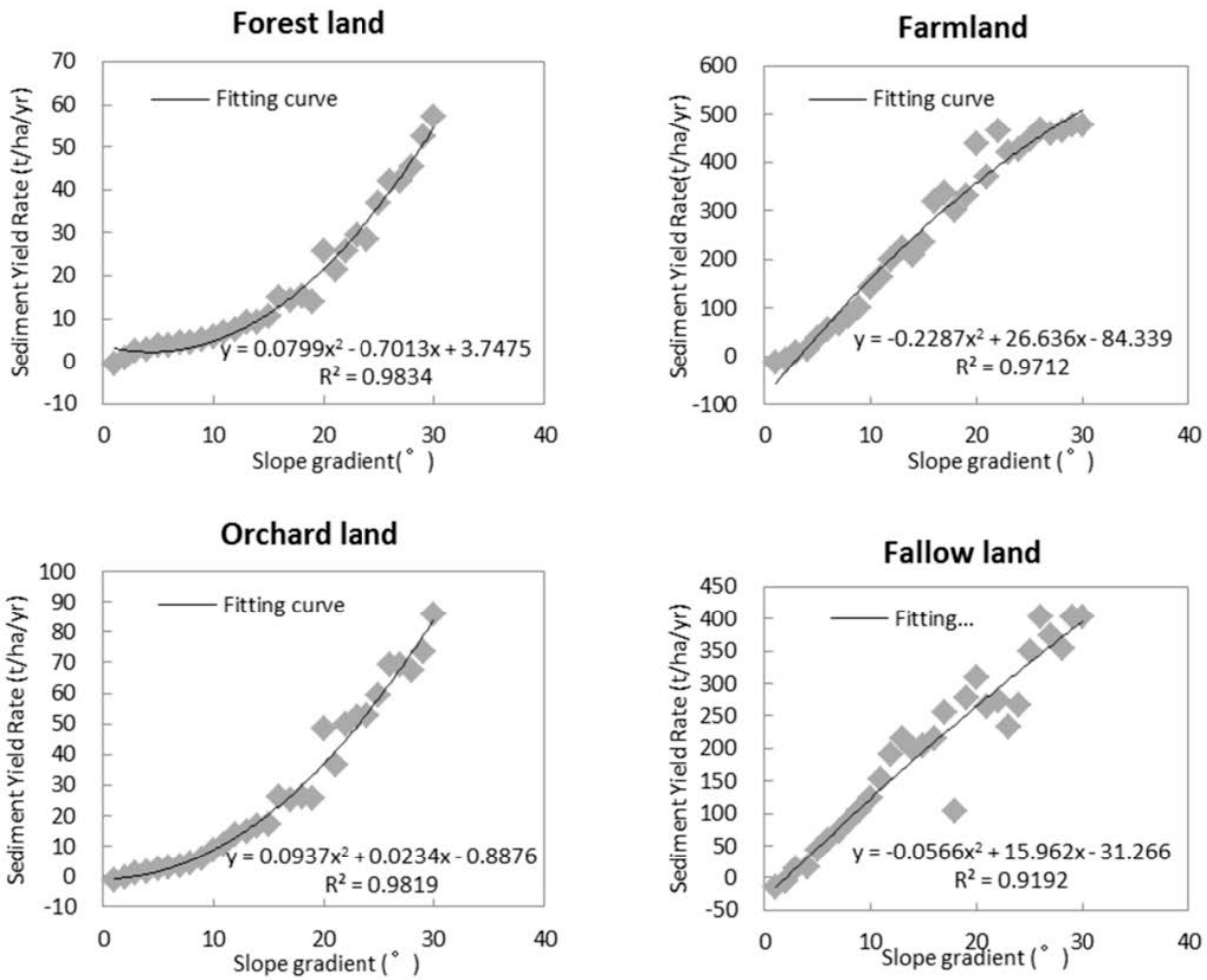

Figure 2.1.4.1 Dependence of sediment yield on slope gradient by land use classes in Jiangxi province, Southern China (Zhang et al., 2015)

Principle

The slope along a line in the surface is expressed either as a percentage, or the number of metres of change in elevation over a horizontal distance of 100 m, or as an angle in degrees, as the measurement of the vertical angle made by the slope and the horizontal plane, or as a ratio of vertical distance to horizontal distance (used to express the slope for the sides of dykes and canals).

Without a map available, slope gradient is to be determined by direct field measurements using an Abney level, a clinometer (OMAFRA, 2016) or a theodolite. Applications for slope measurement exist for smartphones. To ensure accuracy, however, smartphone results must be tested and compared to those from standard measuring procedures. Slope length can simply be measured with a measuring tape.

Reagents

- None

Equipment

- *Abney level:* a protractor is coupled to a sighting tube, with a bubble level and a mirror prism (OMAFRA, 2016). Sliding the eyepiece forward or backward the bubble image is focused. The scale plate (protractor)

has both percent and degree scale graduations. The indicator or scale-pointing arm has a vernier scale. The bubble level on the main body is used to level the instrument.

- *Indian pattern clinometer:* consists of a base plate with a small bubble tube and a leveling screw; the eye vane has a peep hole on the base plate; the object vane has graduations in degrees on one side and the tangent of the angles on the other (OMAFRA, 2016).
- *Buriel hand level:* consists of a frame with a mirror and a plain glass. The principle is that a horizontal ray of light is reflected back from a vertical mirror. With the instrument at eye level, the image of the eye is visible at the edge, while the objects appearing opposite the image of the eye are at the observer's eye level.
- *Foot rule clinometer:* consists of a box wood rule with two arms hinged to each other, both supplied with a small bubble tube, a pair of sights and a graduated arc.
- *Fennel's clinometer:* consists of a telescope, two plate levels, a vertical arc which rotates or tilts with the telescope, and a holding staff with a target.
- *De Lisle's clinometer:* consists of a simple frame with a mirror (vertical reference line) and a semicircular graduated arc with a moveable radial arm.
- *Sextant:* The arrangement of two mirrors enables the observer to sight at two different objects simultaneously. The angle between the mirrors is equal to half the actual angle between two objects. Slope angle can be measured in a single observation.
- *Theodolite:* A movable telescope mounted within two perpendicular axes: the horizontal or trunnion axis and the zenith axis. For measuring vertical angles between the zenith and the ground surface.

Procedure

1. Measurement of vertical angle using Abney level:

1.a. Set the instrument at eye level
1.b. Direct it to the object
1.c. Bring the bubble to the centre
1.d. Read angle on the arc by means of the vernier scale

2. Measurement using the Indian pattern clinometer:

2.a. Set the plane table over the station and keep the clinometer on it
2.b. Use the levelling srew to level the clinometer
2.c. Look through the peep hole, move the slide of the object vane until it bisects the signal
2.d. Read the tangent of the angle
2.e. Calculate the angle from distance x tangent of vertical angle (d tanα)

3. Measurement with the foot rule clinometer:

3.a. Hold the instrument firmly against a rod, with the bubble centred in the lower arm
3.b. Raise the upper arm until the sight line passes through the object
3.c. Take the reading on the arc

4. Measurement with Fennel's clinometer:

4.a. Incline the telescope towards the sighted object
4.b. Make the reading on the diaphragm with stadia lines

5. Measurement with De Lisle's clinometer:

5.a. Slide the weight to the inner stop of the arm
5.b. Turn the arc forward for rising gradients and backwards for falling gradients
5.c. Suspend the instrument and hold it at arm's length to see the reflected image of one's eye at the edge of the mirror
5.d. Move the radial arm until the object sighted is coincident with the reflection of the eye
5.e. Make the reading on the arc

6. Measurement of vertical angle with theodolite:

6.a. Rotate the theodolite until the arrow in the rough sights is lined up with the point to measure
6.b. Look through the small eyepiece, and adjust the knob to obtain a precise horizontal lined up with your object
6.c. Read the slope angle through the small eyepiece, do the vertical measurement

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5. Slope exposure

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

Although slope exposure or aspect is of minor importance in the tropics, it is a major factor influencing the distribution of solar radiation at middle and high latitudes (Chang, 2015). Direct insolation is a function of slope exposure. The indirect effects of slope exposure are associated with the position relative to the direction of the prevailing wind. Soils on windward slopes will typically be shallower, while on leeward slopes winds promote soil formation through depositing fine air-borne particles. At mid-latitudes southwestern slopes (which receive the highest amount of insolation in early afternoon) usually show the lowest soil moisture and lowest soil organic matter content. Slope exposure also influences seasonal temperature-dependent soil biological processes (Bardelli et al., 2017). Under climates with warm or hot summers and cold winters, eastern, northern, and northeastern slopes are the preferred sites for crop cultivation. Southern and western exposures are warmer than eastern and northern exposures. Southern exposures warm earlier in the spring and can slightly advance bud break, thus increasing the potential for frost damage (Stafne, 2015). Eastern slopes are exposed to the morning sun; vinestocks and fruit trees there will dry (from dew or rain) sooner than those on a western slope, potentially reducing disease risk. Management costs can be reduced if the optimal slopes are selected for cultivation.

Principle

Slope exposure/aspect is defined as the directional component of the slope gradient vector, i.e. the direction of maximum gradient of the surface at a given point (FAO, 2006). It is expressed as the compass direction the slope faces (north, south, east or west). It is also often expedient to distinguish secondary directions (northeast, southeast, southwest, northwest). As a GIS derivative, ArcGIS uses Horn's 8-point formula (Burrough & McDonell 1998; De Smith et al., 2018) and slope exposure/aspect is calculated counterclockwise from east (Fig. 2.1.5.1).

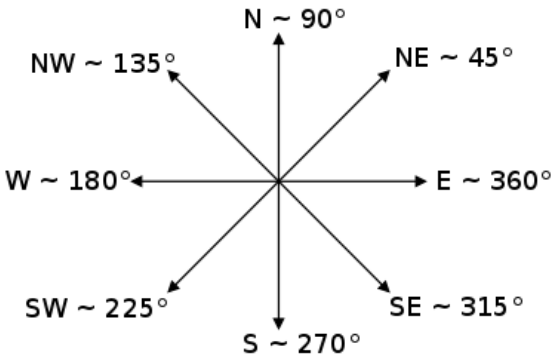

Figure 2.1.5.1 Slope aspects (after Shapiro & Waupotitsch, 2015)

Reagents

- None

Materials and equipment

- None

Procedure

a. Set up a 3 x 3 moving window grid over the studied surface

a	b	c
d	e	f
g	h	i

b. Calculate gradient in different directions for different cells (see Calculations)

Calculations

a. Calculate the rate of change in the **x** direction for cell **e**:

$$[dz/dx] = ((c + 2f + i) - (a + 2d + g)) / 8 \quad \text{(Eq. 2.1.5.1)}$$

b. Calculate the rate of change in the **y** direction for cell **e**:

$$[dz/dy] = ((g + 2h + i) - (a + 2b + c)) / 8 \quad \text{(Eq. 2.1.5.2)}$$

c. Calculate the aspect for cell **e**:

$$aspect = 57.29578 * atan2 ([dz/dy], -[dz/dx]) \quad \text{(Eq. 2.1.5.3)}$$

Remark

- Exposure values can be transformed to azimuth (0 is north, 90 is east, etc)

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6. Parent material

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary.

Importance and applications

Parent material, composed of primary minerals, is among the five factors of soil development. It determines soil physical properties such as texture, structure, water holding capacity and partly or totally the way of weathering, clay content and, thus, influences soil workability and the opportunities for an alternative cropping system (like minimum or no tillage). The mineralogy of the parent material is mirrored in the soil, it affects the processes of weathering and natural vegetation growth (Anderson, 1998, Augusto et al., 2017, Rafael et al., 2018). Soil parent material is also the basic source of nutrients for microbial life (Sun et al., 2015).

Parent material also affects the chemical properties of soils. Particularly in the case of young soils, releasing nutrients (e.g. phosphorous and potassium) by weathering and controlling rooting depth by rock resistance, the parent material has a major influence on soil fertility. Alluvial soils derived from fluvial deposits, for instance, are rich in plant nutrients but deficient in organic C and N. Vineyards and orchards are cultivated on steep slopes on the exposed rock, with a lack of any soil cover, and, thus, parent sediments have a decisive role.

Principle

The task is to reveal to what extent soil parent material determines the nutrient supply and its limitations. Concerning phosphorus, the relationships between actual P pools of soils and physico-chemical properties (acidity, P richness) of the parent material must be quantified (Rafael et al., 2018).

Reagents

- None

Materials and equipment

- None

Procedure

The parent material classification is based on the FAO SOTER system (FAO, 2006), modified by Schuler et al., (2010). The revised classification system consists of several levels with weighted properties (Table 2.1.6.1):

a. level 1: consolidated or unconsolidated
b. level 2: geochemical properties (e.g. siliceous, carbonaceous, saline)
c. level 3: expression strength of geochemical properties
d. level 4: genetic type (e.g. igneous, metamorphic)
e. level 5: rock name according to IUGS terminology or traditional terms.

Table 2.1.6.1 The revised system of soil parent material classification (after Schuler et al., 2010)

Level2	Level 3	Level 4	Level 5
Level 1: C consolidated			
CS siliceous	**CSA** acid (> 66% SiO_2)	**CSAI** igneous	**CSAI1** quartz rich granitic rock, quartzolite
			CSAI2 aplite (75% SiO_2), rhyolite (74% SiO_2), rhyolitic tuff, alkali feldspar rhyolite (73% SiO_2), quartz latite (73% SiO2), granite (72% SiO_2), monzogranite (72% SiO_2), syenogranite (72% SiO_2), pegmatite (71% SiO_2), alkali feldspar granite (70% SiO_2)
			CSAI3 dacite (68% SiO_2), granodiorite (68% SiO_2), quartz syenite (67% SiO_2)
		CSAM metamorphic	**CSAM1** quartzite (81% SiO_2), siliceous shale, siliceous schist
			CSAM2 spilite (71% SiO_2), migmatite (70% SiO_2), gneiss (69% SiO_2), paragneiss, orthogneiss, psammite (69% SiO_2), meta-felsic rock
			CSAM3 semipelite
		CSAS sedimentary rock	**CSAS1** chert (77% SiO_2), flint, radiolarite, spiculite
			CSAS2 quartz arenite, quartz wacke, sandstone (76% SiO_2), conglomerate (73% SiO_2), breccias consisting of acid rock fragments, fanglomerate, arkose (71% SiO_2), arkosic arenite
			CSAS3 greywacke (66% SiO_2), feldspathic greywacke, arkosic wacke
	CSI intermediate (52-66% SiO_2)	**CSII** igneous	**CSII1** tonalite (65% SiO_2), latite (65% SiO_2), obsidian (65% SiO_2), quartz monzonite (64% SiO_2), syenite (63% SiO_2), trachyte (63% SiO_2), quartz alkali feldspar syenite, quartz alkali fedspar trachyte, quartz diorite, quartz gabbro, quartz anorthosite, foid-bearing syenite/alkali feldspar syenite/trachyte
			CSII2 monzonite (59% SiO_2), monzodiorite (59% SiO_2), benmoreite (58% SiO_2), andesite 58% SiO_2), boninite, diorite (57% SiO_2), monzogabbro (56% SiO_2), keratophyre[2] (56% SiO_2), phonolite (55% SiO_2), kersantite (55% SiO_2), foid-bearing monzonite/diorite/ monzodiorite/monzogabbro
			CSII3 alkali feldspar syenite (54% SiO_2), alkali feldspar trachyte, trachyandesite (52% SiO_2),
		CSIM metamorphic	**CSIM1** pelite (63% SiO_2), slate (63% SiO_2), phyllite (62% SiO_2), hornfels (61% SiO_2), schist (60% SiO_2), mica schist, metamudstone
			CSIM2 granofels (56% SiO_2)
			CSIM3 granulite (53% SiO_2)
		CSIS sedimentary rock	**CSIS1** diamictite (61% SiO_2), tillite
			CSIS2 siltstone (61% SiO_2), claystone (61% SiO_2), mudstone (60 SiO_2)
	CSB basic (45-52% SiO_2)	**CSBI** igneous	**CSBI1** basalt (50% SiO_2), dolerite (50% SiO_2), gabbro (49% SiO_2), anorthosite (49% SiO_2), lamprophyre (48% SiO_2), alkali basalt, tholeiite, diabase, foid-bearing gabbro/ anorthosite
			CSBI2 theralite (46% SiO_2), basanite (46% SiO_2), limburgite (46% SiO_2), pyroxenite (46% SiO_2), tephrite (45% SiO_2), basanite (45% SiO_2)
			CSBM1 amphibolite (50% SiO_2)

Level2	Level 3	Level 4	Level 5
		CSBM metamorphic	**CSBM2** meta-basic rock, meta-mafic rock, greenstone, greenschist, blueschist, spillite
			CSBM3 eclogite (50% SiO_2)
			CSBM4 calc-silicate rock (49% SiO_2)
		CSBS sedimentary rock	**CSBS1** breccia (51% SiO_2)
		CSBA artificial	**CSBA1** acid slag (45-50% SiO_2)
	CSU ultrabasic (<45% SiO_2)	**CSUI** igneous	**CSUI1** foid syenite, foid monzonite, foid monzodiorite, foid monzogabbro, foid diorite, foid gabbro
			CSUI2 leucitite (44% SiO_2), nephelinite (44% SiO_2), foidolite, foidite
			CSUI3 picrite (43% SiO_2), komatiite (41% SiO_2), meimechite
			CSUI4 hornblendite (41% SiO_2)
			CSUI5 peridotite (39% SiO_2)
			CSUI6 melilitite (37% SiO_2)
			CSUI6 kimberlite (29% SiO_2)
		CSUM meta-morphic	**CSUM1** serpentinite (43% SiO_2), meta-ultramafic rock
			CSUM2 skarn (42% SiO_2)
		CSUA artificial	**CSUA1** basic slag (25-30% SiO_2)
	CSX unspecified	**CSXI** igneous	**CSXIx** igneous rock (unspecified)
			CSXI1 agglomerate, pyroclastic breccia, scoria
			CSXI2 tuff-breccia
			CSXI3 lapilli-stone
			CSXI4 lapilli-tuff
			CSXI5 tuff, consolidated ignimbrite (welded tuff)
		CSXM metamorphic	**CSXMx** metamorphic rock (unspecified)
			CSXM1 suevite, impactite, impact-melt breccias, impact-melt rock
			CSXM2 cataclasite, mylonite
		CSXS sedimentary rock	**CSXSx** sedimentary rock (unspecified)
			CSXS1 tuffaceous-sedimentary rock, tuffite
CC carbonatic	**CCP** pure	**CCPM** metamorphic	**CCPM1** marble
		CCPS sedimentary rock	**CCPS1** limestone, travertine
			CCPS2 dolomite
	CCI impure	**CCIS** sedimentary rock	**CCIS1** impure limestone, impure dolomite, marlstone
	CCX unspecified	**CCXI** igneous	**CCXI1** carbonatite
		CCXM metamorphic	**CCXMx** metacarbonate rock
		CCXS sedimentary rock	**CCXSx** carbonatic sedimentary rock (unspecified)
CY saltic	**CYX** unspecified	**CYXS** sedimentary rock	**CYXS1** halite, sylvite
CG gypsic	**CGX** unspecified	**CGXS** sedimentary rock	**CGXS1** gypsum, anhydrite
CP phosphatic	**CPX** unspecified	**CPXS** sedimentary rock	**CPXS1** phosphorite, guano

Level2	Level 3	Level 4	Level 5
CO organic	**COX** unspecified	**COXS** sedimentary rock	**COXS1** bituminous coal, anthracite, graphite
CF fealic	**CFX** unspecified	**CFXS** sedimentary rock	**CFXS1** ironstone, iron ore
Level 1: S semi-consolidated			
SS siliceous	**SSA** acid	**SSAR** residual deposit	**SSAR1** kaolin
SC carbonatic	**SCX** unspecified	**SCXS** sedimentary rock	**SCXS1** chalk
			SCXS2 tufa
SF fealic	**SFX** unspecified	**SFXS** sedimentary rock	**SFXS1** laterite, bauxite
SO organic	**SOX** unspecified	**SOXS** sedimentary rock	**SOXS1** lignite
			SOXS1 asphalt
Level 1: U unconsolidated			
US siliceous	**USA** acid (>66% SiO_2)	**USAI** igneous	**SOXS1** pumice
		USAS sediment	**USAS1** sand (77% SiO_2)
			USAS2 gravel (67% SiO_2)
	USI intermediate (52-66% SiO_2)	**USIS** sediment	**USIS1** silt (57% SiO_2)
			USIS2 clay (59% SiO_2)
	USX unspecified	**USXI** igneous	**USXIx** igneous unconsolidated (unspecified)
			USXI1 block-tephra, bomb-tephra
			USXI2 ash-breccia
			USXI3 lapilli-tephra
			USXI4 lapilli-ash
			USXI5 ash, unconsolidated ignimbrite (non-welded sillar)
		USXS sediment	**USXSx** sediment (unspecified)
			USXS1 breccia
			USXS2 loess
			USXS3 loam
			USXS4 mud, siliceous ooze
			USXS5 diamicton, till
		USXA anthropogenic	**USXA1** waste
			USXA2 heap material
			USXA3 ash (anthropogenic)
			USXA4 brick
			USXA5 mud
UC carbonatic	**UCX** unspecified	**UCXS** sediment	**UCXS1** carbonate sand
			UCXS2 carbonate mud, carbonate ooze
			UCXS3 carbonatic diamicton
			UCXS4 carbonatic sediment, marl
		UCXA anthropogenic	**UCXA1** lime plaster, cement plaster
			UCXA2 concrete
			UCXA3 waste combustion ash
UO organic	**UOX** unspecified	**UOXS** sediment	**UOXS1** half-bog
			UOXS2 peat

Level2	Level 3	Level 4	Level 5
			UOXS3 sapropel
		UOXA anthropogenic	**UOXA1** plaggen
			UOXS2 coal/coke dump material
			UOXS3 road construction material: tar, asphalt, bitumen)
UY saltic	**UYX** unspecified	**UYXS** sediment	**UYXS1** salt mud
		UYXA anthropogenic	**UYXS2** saline material
UG gypsic	**UGX** unspecified	**UGXS** sediment	**UGXS1** gypsum-mud
		UGXA anthropogenic	**UGXA1** gypsum plaster
UP phosphatic	**UPX** unspecified	**UPXS** sediment	**UPXS1** phosphoric mud
UF fealic	**UFX** unspecified	**UFXS** sediment	**UFXS1** iron sediment
		UFXA anthropogenic	**UFXA1** red mud
			UFXA2 metal-sludge
UR radioactive	**URX** unspecified	**URXA** anthropogenic	**URXA1** nuclear waste
X unspecified	**X** unspecified	**X** unspecified	**X** unspecified

E	**X**	**X**	**x** evaporitic rock sequence
K	**X**	**X**	**x** carbonatic rock sequence
L	**X**	**X**	**x** organic rock sequence
M	**X**	**X**	**x** iron ore sequence

Remarks

- The influence of parent material on soil properties is usually indirect and, therefore, difficult to detect.
- Multiple parent materials can be found in many soil profiles. For instance, the conditions of soil formation will be fundamentally different in those with thin slope deposits or loess covering the volcanic bedrock, from those on bare bedrock.

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7. Type of soil horizon

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

The diagnostic horizons of soil taxonomy are not the same as those used in the genetic soil classification: the designations of genetic horizons are based on a qualitative judgement on soil formation, diagnostic horizons are quantitatively defined. A diagnostic horizon may encompass several genetic horizons (Soil Survey Staff, 2014). In the World Reference Base (WRB) such diagnostic horizons serve as a basis for classification (FAO, 2015). The WRB soil classification is widely applied in designing sustainable soil management techniques (Lal & Stewart, 2013; Stolte et al., 2016).

Principle

Soil horizons must be clearly defined and designated in any soil classification. The classification of soils is based on diagnostic horizons, diagnostic properties and diagnostic materials (FAO, 2015). A soil horizon is a unit of mineral or organic soil material approximately parallel to the land surface with properties altered by soil formation processes, different from adjacent horizons in colour, texture, structure, consistency as well as chemical, biological or mineralogical composition (Agriculture Canada, 2013). The major mineral horizons are A, B, and C. The major organic horizons are L, F, and H (decomposed forest litter) and O (wetland organic matter). Subclasses are identified in the field or in the laboratory by adding lower-case suffixes to the main horizon symbols.

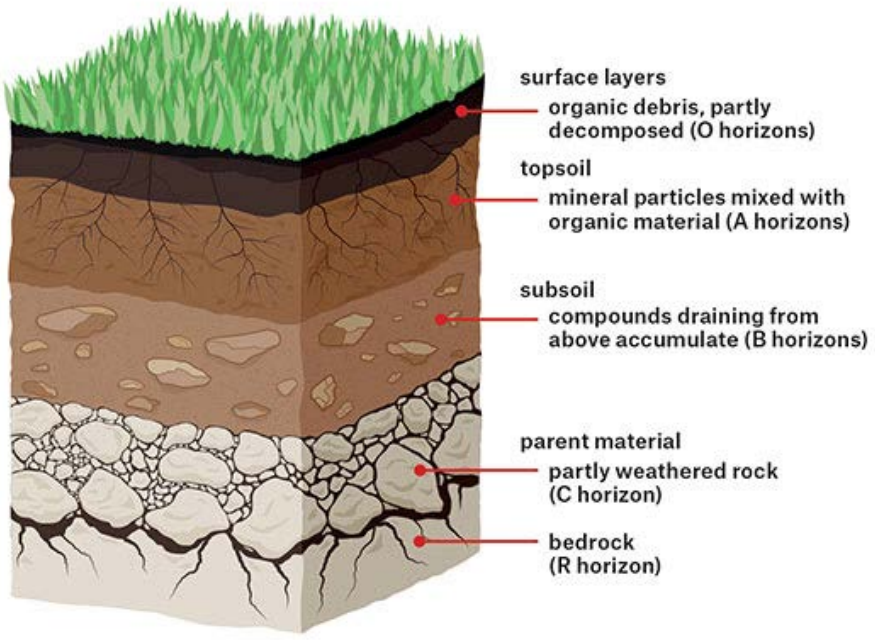

Figure 2.1.7.1 Master soil horizons

Material and equipment

The identification of soil horizons is based on bulk density, particle size distribution, Munsell colour, soil reaction, organic matter and carbonate contents etc., for materials and so see the relevant chapters.

Procedure

a. Determine bulk density, particle size distribution, Munsell colour, soil reaction, organic matter and carbonate contents etc. for the soil horizon
b. Determine master soil horizon
c. Use Table 2.1.7.1 to identify diagnostic horizon type

Table 2.1.7.1 The diagnostic horizons, properties and materials of the WRB (FAO, 2015)

1. **Anthropogenic diagnostic horizons** (all are mineral)

- *anthraquic horizon* in paddy soils: the layer comprising the puddled layer and the plough pan, both showing a reduced matrix and oxidised root channels
- *hortic horizon*: dark, high content of organic matter and P, high animal activity, high base saturation; resulting from long-term cultivation, fertilisation and application of organic residues
- *hydragric horizon* in paddy soils: the layer below the anthraquic horizon showing redoximorphic features and/or an accumulation of Fe and/or Mn
- *irragric horizon*: uniformly structured, at least moderate content of organic matter, high animal activity; gradually built up by sediment-rich irrigation water
- *plaggic horizon*: dark, at least moderate content of organic matter, sandy or loamy; resulting from application of sods and excrements
- *pretic horizon*: dark, high content of organic matter and P, low animal activity, high contents of exchangeable Ca and Mg, with remnants of charcoal and/or artefacts; including Amazonian Dark Earths
- *terric horizon*: showing a colour related the source material, high base saturation; resulting from adding mineral material (with or without organic residues) and deep cultivation

2. **Diagnostic horizons that may be organic or mineral**

- *cryic horizon*: perennially frozen (visible ice or, if not enough water, ≤ 0°C)
- *calcic horizon*: accumulation of secondary carbonates, non-cemented
- *fulvic horizon*: andic properties, highly humified organic matter, higher ratio of fulvic acids to humic acids
- *melanic horizon*: andic properties, highly humified organic matter, lower ratio of fulvic acids to humic acids, blackish
- *salic horizon*: high amounts of readily soluble salts
- *thionic horizon*: with sulphuric acid and a very low pH

3. **Organic diagnostic horizons**

- *folic horizon*: organic layer, not water-saturated and not drained
- *histic horizon*: organic layer, water-saturated or drained

4. **Surface mineral diagnostic horizons**

- *chernic horizon*: thick, very dark-coloured, high base saturation, moderate to high content of organic matter, well-structured, high biological activity (special case of the mollic horizon)
- *mollic horizon*: thick, dark-coloured, high base saturation, moderate to high content of organic matter, not massive and hard when dry
- *umbric horizon*: thick, dark-coloured, low base saturation, moderate to high content of organic matter, not massive and hard when dry

5. Other mineral diagnostic horizons related to the accumulation of substances due to (vertical or lateral) migration processes

- *argic horizon*: subsurface layer with distinctly higher clay content than the overlying layer and/or presence of illuvial clay
- *duric horizon*: concretions or nodules, cemented or indurated by silica
- *ferric horizon*: ≥ 5% reddish to blackish concretions and/or nodules or ≥15 % reddish to blackish coarse mottles, with accumulation of Fe (and Mn) oxides
- *gypsic horizon*: accumulation of secondary gypsum, non-cemented
- *natric horizon*: subsurface layer with distinctly higher clay content than the overlying layer and/or presence of illuvial clay; high content of exchangeable Na
- *petrocalcic horizon*: accumulation of secondary carbonates, relatively continuously cemented or indurated
- *petroduric horizon*: accumulation of secondary silica, relatively continuously cemented or indurated
- *petrogypsic horizon*: accumulation of secondary gypsum, relatively continuously cemented or indurated
- *petroplinthic horizon*: sheet of connected yellowish, reddish and/or blackish concretions and/or nodules or of concentrations in platy, polygonal or reticulate patterns; high contents of Fe oxides at least in the concretions, nodules or concentrations; relatively continuously cemented or indurated
- *pisoplinthic horizon*: ≥ 40% strongly cemented to indurated, yellowish, reddish, and/or blackish concretions and/or nodules, with accumulation of Fe oxides
- *plinthic horizon*: ≥ 15% (single or in combination) of reddish concretions and/or nodules or of concentrations in platy, polygonal or reticulate patterns; high contents of Fe oxides, at least in the concretions, nodules or concentrations
- *sombric horizon*: subsurface accumulation of organic matter other than in spodic or natric horizons
- *spodic horizon*: subsurface accumulation of organic matter and/or Fe and Al

6. Other mineral diagnostic horizons

- *cambic horizon*: evidence of pedogenic alteration; not meeting the criteria of diagnostic horizons that indicate stronger alteration or accumulation processes
- *ferralic horizon*: strongly weathered; dominated by kaolinites and oxides
- *fragic horizon*: structure compact to the extent that roots and percolating water penetrate only along interped faces; non-cemented
- *nitic horizon*: rich in clay and Fe oxides, moderate to strong structure, shiny aggregate faces
- *protovertic horizon*: influenced by swelling and shrinking clays
- *vertic horizon*: dominated by swelling and shrinking clays

7. Diagnostic properties related to surface characteristics

- *aridic properties*: surface layer characteristics of soils under arid conditions

- *takyric properties*: heavy-textured surface layers under arid conditions in periodically flooded soils (special case of aridic properties)
- *yermic properties*: pavement and/or vesicular layer in soils under arid conditions (special case of aridic properties)

8. Diagnostic properties defining the relationship between two layers

- abrupt textural difference
- very sharp increase in clay content within a limited depth range
- albeluvic glossae interfingering of coarser-textured and lighter coloured material into an argic horizon forming vertically continuous tongues (special case of retic properties)
- lithic discontinuity differences in parent material
- retic properties interfingering of coarser-textured and lighter coloured material into an argic or natric horizon

9. Other diagnostic properties

- *andic properties*: short-range-order minerals and/or organo-metallic complexes
- *anthric properties*: applying to soils with mollic or umbric horizons, if the mollic or umbric horizon is created or substantially transformed by humans
- continuous rock consolidated material (excluding cemented or indurated pedogenetic horizons)
- *geric properties*: very low effective CEC and/or acting as anion exchanger
- *gleyic properties*: saturated with groundwater (or upwards moving gases) long enough for reducing conditions to occur
- *protocalcic properties*: carbonates derived from the soil solution and precipitated in the soil (secondary carbonates), less pronounced than in calcic or petrocalcic horizons
- *reducing conditions*: low pH value and/or presence of sulphide, methane or reduced Fe
- shrink-swell cracks open and close due to swelling and shrinking of clay minerals
- *sideralic properties*: relatively low CEC
- *stagnic properties*: saturated with surface water (or intruding liquids), at least temporarily, long enough for reducing conditions to occur, vitric properties ≥ 5% (by grain count) of volcanic glass and related materials and containing a limited amount of short-range-order minerals and/or organo-metallic complexes

10. Diagnostic materials related to the concentration of organic carbon

- *mineral material*: < 20% soil organic carbon
- *organic material*: ≥ 20% soil organic carbon
- *soil organic carbon*: organic carbon that does not meet the diagnostic criteria of artefacts

11. Diagnostic material related to colour

- *albic material*: light-coloured fine earth, expressed by high Munsell value and low chroma

12. Technogenic diagnostic materials (predominantly understood as parent materials)

- artefacts created, substantially modified or brought to the surface by humans; no subsequent substantial change of chemical or mineralogical properties
- *technic hard material*: consolidated and relatively continuous material resulting from an industrial process

13. **Other diagnostic materials** (predominantly understood as parent materials)

- *calcaric material*: ≥ 2% calcium carbonate equivalent, inherited from the parent material
- *colluvic material*: heterogeneous mixture that has moved down a slope
- *dolomitic material*: ≥ 2% of a mineral that has a ratio $CaCO_3/MgCO_3 < 1.5$
- *fluvic material*: fluviatile, marine or lacustrine deposits with evident stratification
- *gypsiric material*: ≥ 5% gypsum, at least partially inherited from the parent material
- *hypersulphidic material*: sulphidic material capable of severe acidification
- *hyposulphidic material*: sulphidic material not capable of severe acidification
- *limnic material*: deposited in water by precipitation or through action of aquatic organisms
- *ornithogenic material*: remnants of birds or bird activity
- *sulphidic material*: containing detectable inorganic sulphides
- *tephric material*: ≥ 30% (by grain count) of volcanic glass and related materials

Remarks

- All horizons may be vertically subdivided by consecutive numeral suffixes, e.g. A_{e1} and A_{e2}.
- The upper-case horizon designations A, B and O are always accompanied by lower-case specifications, e.g. A_h, B_w, O_m.
- In some cases, such as B_{gf} and B_{hf}, the combination of suffixes does not simply show the sum of the two suffixes used singly.

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8. Depth and thickness of horizon

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

A soil horizon is distinguished from other horizons by texture, colour and structure, which result from soil-forming processes. The individual horizons of a soil profile are of variable thickness. Along with its designation, the depth to and thickness of the horizon should also be recorded because this informs about important ecological properties, such as water-holding and filtering capacity, and rooting depth (Cousin et al., 2009).

Principle

The description of pedons is essential for soil surveys. A pedon is a three-dimensional body of soil that has sufficient area (roughly 1 to 10 m^2) and depth (up to 200 cm). When describing the sequence of horizons, quantitative and qualitative data are equally used for the description of the individual soil horizons. The depth to the lower boundary of a horizon is always the depth to the upper boundary of the underlying horizon. The thickness of each horizon or layer is the vertical distance between the upper and lower boundaries.

Reagents

- None

Materials and equipment

- Measuring tape

Procedure

a. Dig soil pit
b. Clean all sides of the pit of all loose material disturbed by digging
c. Measure the upper and lower boundaries of each horizon on exposed vertical faces
d. Take photographs of all horizons identified

Calculations

- None

Remarks

- The accuracy required for the determination of soil horizon depth and thickness does not usually exceed 5–10 cm.

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9. Bulk density

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

Bulk density (D_b) is important both for soil description (evaluation of soil fertility) and the provision of ecosystem services. Measurements of physical properties such as bulk density and pore size distribution are relevant to assess soil compaction at the macroscopic scale (Gupta et al., 1989). High bulk density is a limiting factor of root penetration (Table 2.1.9.1), soil aeration and water infiltration (FAO, 2006).

Resulting from bad management practices, soil compaction causes an increase in soil bulk density. Porosity is reduced in the compacted layer which loses its ability to transmit water. The compaction layer may result in perched water tables and waterlogging. In a dry state, the compacted layer is a physical barrier to root growth, restricts rooting depth and limits the availability of water and nutrients to the crop (Moody & Cong, 2008).

Table 2.1.9.1 Relationship between bulk density and root growth

Soil texture	Ideal bulk density (g cm^{-3})	Marginal bulk density (g cm^{-3})	Root restricting bulk density (g cm^{-3})
Sands, loamy sands	< 1.60	1.69	> 1.80
Sandy loams, loams	< 1.40	1.63	> 1.80
Sandy clay loams, clay loams	< 1.40	1.60	> 1.75
Silts, silt loams	< 1.40	1.60	> 1.75
Silt loams, silty clay loams	< 1.40	1.55	> 1.65
Sandy clays, silty clays, loams, clay loams	< 1.10	1.49	> 1.58
Clays (> 45% clay)	< 1.10	1.39	> 1.47

Principle

Bulk density is defined as the mass of soil (M_{solids}) of unit volume (V_{soil}) in a dry state, i.e. at 105°C temperature. Thus, the bulk density reflects total soil porosity (FAO, 2006). Total soil volume is the volume of solids and pores together: both pore air (V_{air}) and water volume (V_{water}). If values are low (generally below the threshold of 1.3–1.6 g cm^{-3}), porosity is high. There are several methods for determining soil bulk density. Field estimation of bulk density refers to the force required to push a knife into a soil horizon exposed at a field moist pit wall. Three sampling methods are common: the core, the excavation and the clod method (ISO, 2017). Here we report the core method by digging a ring in the soil.

Reagents

- None

Materials and equipment

- Bulk Density Sampler Cup and Cap
- Cylinder, metal ring (100-500 cm^3)
- Oven
- Scale (accuracy: 0.01 g)
- (For the coat clod method): paraffin or other water-repellent substance

Procedure

a. Remove the cup of ring
b. Dry undisturbed soil sample, drying at 105°C 24 h
c. Scale m_2 (metal ring + soil)
d. For surface horizons, a simple excavation method is applied:
e. Dig a soil pit
f. Fill it completely with a measured volume of sand
g. The clod method is used in cases of large soil aggregates, with the help of paraffin or other water-repellent substance coatings (Hirmas & Furquim, 2006)
h. Weigh the coated clod in air
i. Measure the volume of water displaced by the clod in a graduated cylinder
j. Wash the paraffin-coated clod in boiling water to separate the paraffin from gravel and hardened soil aggregates
k. Weigh the clod in water to determine its volume

Calculations

$$\varrho_b = (m_2-m_1)v^{-1} \quad [M\ L^{-3};\ g\ cm^{-3};\ kg\ m^{-3};\ Mg\ m^{-3}] \qquad \text{(Eq: 2.1.9.2)}$$

where

ϱ_b is the bulk density of the soil [$g\ m^{-3}$],
m_1 is the weight of the metal ring [g],
m_2 is the weight of the metal ring + soil after drying [g],
V is the volume of the metal ring [cm^{-3}].

Remarks

- In the case of soils with gravels and boulders (Regosols, Mixed Anthrosol) coarse constituents hinder sampling and increase measurement error
- In the case of soils with swelling clay minerals correction calculation is needed related to the field volume/dry volume ratio

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10. Particle size distribution

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

Important soil properties are associated with particle size distribution (PSD), among others: porosity, permeability, infiltration, shrinking-swelling, water-holding capacity, susceptibility to erosion and compaction, organic matter dynamics. PSD controls the rate of drainage of a saturated soil. Water percolates relatively freely through sandy soils. Clayey soils have higher water-holding capacities than sandy soils. Well-drained soils show good aeration and contain air similar to atmospheric air. Soil textures also differ in their susceptibility to erosion (erodibility): those with a high percentage of silt and clay are of higher erodibility than sandy soils. Organic matter breaks down more rapidly in sandy soils if environmental conditions are otherwise the same. Tillage and soil management are also influenced by particle size proportions: in lighter-textured soils more oxygen is available for decomposition. The cation exchange capacity of the soil grows with increased clay and organic matter percentage.

Principle

PSD involves the determination of the percentages of different grain size classes in a soil. There are two principal approaches (Taubner *et al*., 2009): mechanical or sieve analysis (for the coarser, larger-sized particles) and the hydrometer and/or laser-beam based methods (for the finer particles). *Sieving* is the first step to determine PSD if the soil contains coarse sands, gravels or pebbles. The pipette method explained in this chapter depends on the assumption that sedimentation eliminates from the depth L [m], in a time t [sec], all the particles having settling velocities greater than $L\ t^{-1}$, while retaining at that depth the original concentration of particles with settling velocities less than $L\ t^{-1}$.

In the system of the United States Department of Agriculture (Soil Survey Staff, 1975) and that of the FAO (2006), the particle-size classes are named similarly (Table 2.1.10.1) and the textural class of a soil is defined by use of textural diagrams (Figure 2.1.10.1) to the common standard terminology i (Table 2.1.10.2. The phi scale is widely used in the statistical analyses: $phi=\log_2 D$ [D in mm] (Krumbein, 1938).

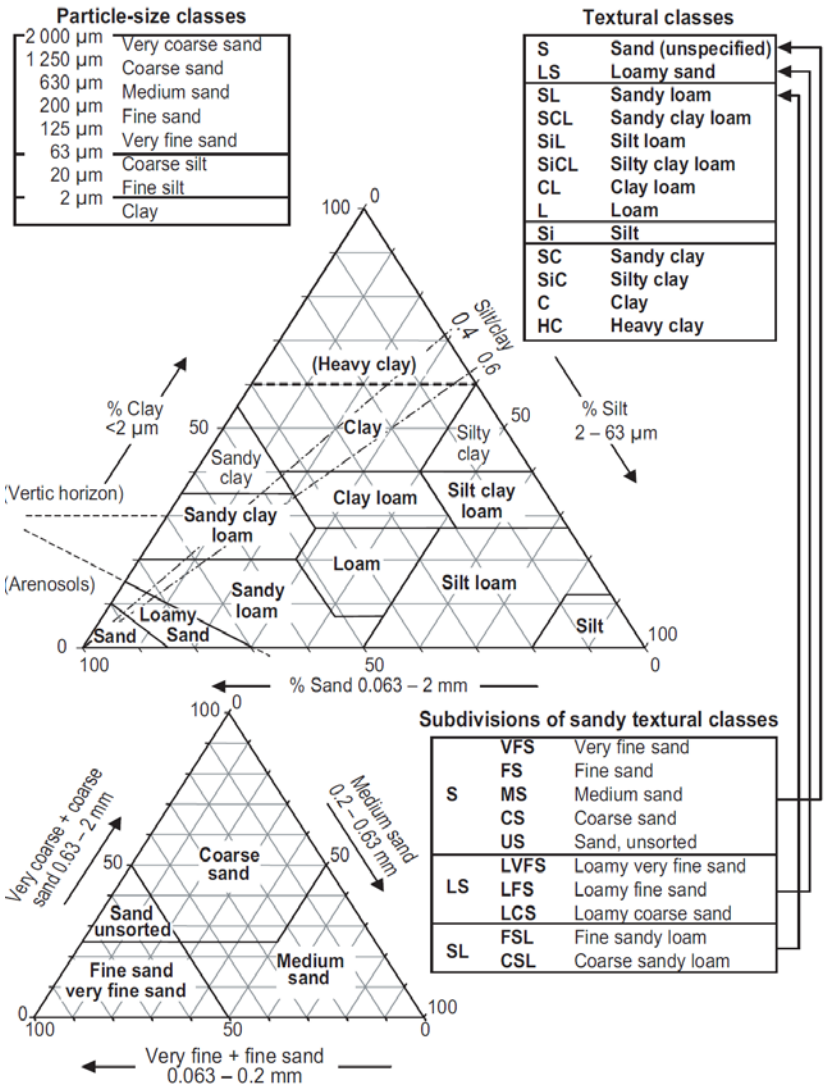

Figure 2.1.10.1 Assessment of textural classes (FAO, 2006). HC = heavy clay; C = clay; L = loam; Si = silt; S = sand

Table 2.1.10.2 Particle size classes (FAO, 2006) and the methods used

Phi	Sieve/particle diameter [µm]	Range [mm-mm]	Categories	FAO 2006	Method		
-6	64,000	> 64	Very coarse pebbles	Stones (60.00–200.00 mm)	Sieving		
-5	32,000	32–64	Very coarse pebbles	Coarse gravel (20.00–60.00 mm)			
-4	16,000	16–32	Coarse pebbles				
-3	8,000	8–16	Medium pebbles	Medium gravel (6.00–20.00 mm)			
-2	4,000	4–8	Fine pebbles	Fine gravel (2.00–6.00 mm)			
-1	2,000	2–4	Very fine pebbles				
0	1,000	1–2	Very coarse sand	Very coarse sand (1.250–2.00 mm)			Laser (MALVERN Mastersizer 3000 (200 nm–3,000 µm))
1	500	0.50–1	Coarse sand	Coarse sand (0.630–1.250 mm)			
2	250	0.250–0.50	Medium sand	Medium sand (0.200–0.630 mm)			
3	0.125	0.125–0.250	Fine sand	Fine sand (0.125–0.200)		Laser (FRITSCH Analysette A22-32)	
4	0.063	0.063–0.125	Very fine sand	Very fine sand	Pipette		
5	0.031	0.031–0.063	Very coarse silt	Coarse silt (0.020–0.063)			
6	0.016	0.016–0.031	Coarse silt				
7	0.08	0.008–0.016	Medium silt	Fine silt (0.002–0.020)			
8	0.04	0.004–008	Fine silt				
9	0.002	0.002–0.004	Very fine silt				
10	0.001	0.001–0.002	Clay	Clay (0.000–0.002)			
11	0.000–0.001	0.000– 0.001	Fine clay + colloids				

Reagents

- 30% H_2O_2, to remove organic material
- 10% HCl to remove carbonates
- Dithionite-Citrate system with 1M $NaHCO_3$ to remove iron-oxides (Mehra & Jackson, 1960)

- Sodium-acetate (1M), or sodium-hexametaphosphate (Calgon) to remove/eliminate the calcium and magnesium ions from the solution

Materials and equipment

For sieving:

- Scale
- Set of sieves (according to the estimated range of particle size),
- Automatic sieve shaker

For pipette method:

- Electric mixer and cup
- Sedimentation cylinder (1,000 mL)
- 20-ml pipette with attached suction bulb
- Oven
- Glass beakers

For laser particle sizer:

- Laser-beam based equipment

Procedure

For sieving:

1. *Sieve Analysis*

1.a. Weigh 100 g (clay); 200 g (silt), 500 g (coarse sand), 1,000 g (gravel), 2,000 g

1.b. Heat the soil sample to 105°C in an oven and keep for 24 hours

1.c. Fix the sieve series on the sieve shaker. The size of the sieves (sieve column) depends on the range of particle distribution and the predictable number of sieves (see section on sieve sizes and classes)

1.2. Wet sieving:

1.2.a. Treat the soil sample with 30% H_2O_2, to remove organic material; 10% HCl, Sodium Pyrophosphate ($Na_4P_2O_7$) or Calgon to remove carbonates, Dithionite-Citrate-Sodium Bicarbonate to remove iron-oxides if needed. Continue the treatment until the soil particles reach a suspended phase

1.2.b. Pour it on the sieve series and close the series with a cap to fix the sieve column

1.2.c. Set the appropriate vibration intensity and time, launch the sieve-shaker. If the sieve-shaker is constructed by a pump for circulating the sieved material, set and control the pump yield. Check fouling in case of a high percentage of fine material

1.2.d. At the end of sieving take the sieve series apart

1.2.e. Remove the soil fractions from the sieves. Do so cautiously to avoid loss of material

1.2.f. Put the wet material into the oven and dry it at 105°C for 24 hours

1.3. Dry sieving

1.3.a. Take the dried sample on the sieve column

1.3.b. Set the appropriate vibration intensity and time, launch the sieve-shaker

1.3.c. At the end of sieving take the sieve series apart

1.3.d. Remove the soil fractions from the sieves

1.3.e. Measure the soil fractions

2. *For the pipette method:*

2.a. Transfer a prepared sample to a 1,000 cm^3 graduated cylinder

2.b. Mix the sample with a plunger or by inversion for one minute or until homogenised

2.c. About 20 seconds is allowed to pass before drawing the initial aliquot to permit a reduction in turbulence

2.d. Remove the initial aliquot using a 20-mL pipette with attached suction bulb

2.e. Proceed in a similar fashion for the remainder of the aliquots at times and depths

2.f. Dry the aliquots to a constant weight in an oven at 90^oC

3. *For laser analyser:*

Based on the Mie-Fraunhoffer method. The setting and procedure strongly depends on manual options on the particle sizer.

3.a. Set the calculation parmeters: interpolation values as oversize or undersize

3.b. Set the mode of measurement: wet or dry method

3.c. Interpolation values, fixed particle sizes, oversize or undersize µm

3.d. Calculation form: cummulative and/or differential calculation

3.e. Distribution forms: model independent, monomodal etc.

3.f. Set measuring range and channels which generates the resolution

3.g. Beam obscuration percentage (generally: 8%)

3.h. Number of measurements = “scan” (3 times recommended)

3.i. Set the graphical mode of the result (triangle, Q-distribution, Gauss distribution, etc)

3.j. Set the raw data file type for exporting (text file recommended)

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11. Munsell colour

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

Soil horizons have different colours, reflecting chemical processes acting on the soil (weathering, oxidation-reduction of minerals, particularly iron and manganese minerals and the decomposition of organic matter). The colour indicates properties that are important for soil management: texture, moisture and organic matter content. For the purposes of soil classification, the Munsell system allows for direct comparison of soils anywhere in the world.

Principle

The Munsell system is a colour system that is based on three dimensions: hue, value and chroma (Fig. 2.1.11.1). The **hue** of a color indicates how it relates to the 'pure' colours red, yellow, green, blue and purple, i.e. what the predominant wavelength of the reflected light is. **Value** denotes lightness or darkness. A value of 0 is black and 10 is pure white. **Chroma** marks colour saturation (intensity). A chroma of 0 is neutral grey and the maximum chroma is 20 (but it is never approached). Value becomes successively lighter vertically, from the bottom upward, by visually equal steps. Chroma increases horizontally to the right and becomes greyer to the left.

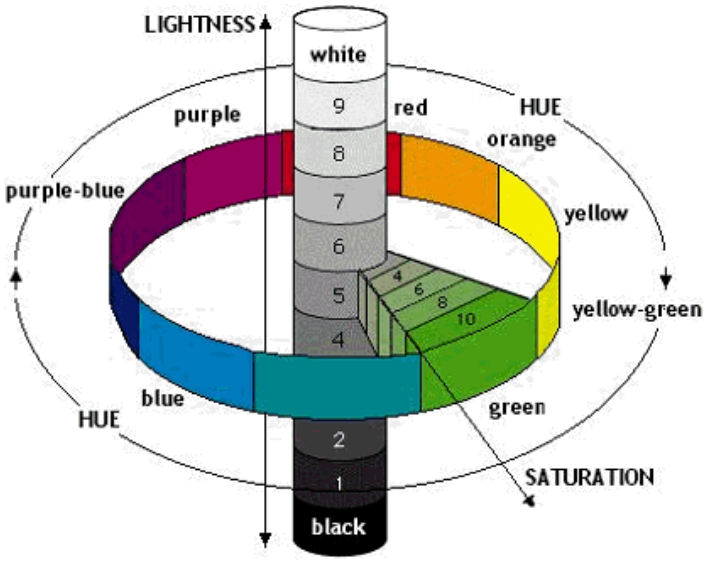

Figure 2.1.11.1 The Munsell colour system

Reagents

- Water for set moist condition of the soil

Materials and equipment

- Munsell Soil Color Charts

Procedure

a. Create a homogeneous soil aggregate under semi-wet conditions
b. Fit the aggregate by visual comparison to the Munsell Color Chart
c. Read hue, value and chroma codes

Remarks

- In soils a mottling pattern may refer to soil aeration or drainage. Mottles (spots in the soil matrix) are of genetic importance and differ in colour from the matrix and their colour should be determined separately
- In mixed soil profiles the colours of the matrix and the enclosed clasts have to be determined

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12. Soil reaction (pH)

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

Soil reaction is important because it affects nutrient availability, microbial activity and plant growth. Most plant species perform best in a pH range 5.5 to 6.5 or 7.0 although some prefer extremes (Table 2.1.12.1). Soil pH controls the solubility of nutrients and, thus, their availability for plants (Fig. 2.1.12.1). In humid climates the leaching of calcium, magnesium, potassium and sodium ions naturally causes a decrease in pH over time because it leaves the soil clays dominated by H+ and aluminium ions (Al^{3+}) (Ketterings et al., 2005). Human activity also influences soil pH. Applying nitrogen fertilisers, manure or compost, sources of organic nutrients, nitric acid (HNO_3) and/or sulphuric acid (H_2SO_4), which are strong acids, form soil acidity (Foth & Ellis, 1997).

Table 2.1.12.1 Preferred pH ranges for common crops (Albert, 2018)

Acidophile crops (pH from 4 to 5.5)	Slightly acidophile crops (tolerate pH from 5.5 to 6.5)	Moderately alkalophile crops (tolerate pH from 6.0 to 7.0 or greater)		Crops of great tolerance: (tolerate a wide range of soil acidity or alkalinity, from about 5.0 to 7.0)
Blackberry (5.0–6.0)	Apple (5.0–6.5)	Artichoke (6.5–7.5)	Jerusalem Artichoke/ Sunchoke (6.7–7.0)	Alpine strawberry (5.0–7.5)
Blueberry (4.5–5.0)	Basil (5.5–6.5)	Arugula (6.5–7.5)	Kale (6.0–7.5)	Carrot (5.5–7.0)
Cranberry (4.0–5.5)	Carrot (5.5–7.0)	Asparagus (6.0–8.0)	Kohlrabi (6.0–7.5)	Cauliflower (5.5–7.5)
Parsley (5.0–7.0)	Cauliflower (5.5–7.5)	Bean, pole (6.0–7.5)	Leek (6.0–8.0)	Corn (5.5–7.5)
Peanut (5.0–7.5)	Chervil (6.0–6.7)	Bean, lima (6.0–7.0)	Lettuce (6.0–7.0)	Cucumber (5.5–7.0)
Potato (4.5–6.0)	Corn (5.5–7.5.)	Beet (6.0–7.5)	Marjoram (6.0–8.0)	Dill (5.5–6.7)
Raspberry (5.5–6.5)	Cucumber (5.5–7.0)	Broccoli (6.0–7.0)	Mizuna (6.5–7.0)	Endive/Escarole (5.8–7.0)
Sweet potato (5.5–6.0)	Dill (5.5–6.5)	Broccoli rabe (6.5–7.5)	Mustard (6.0–7.5)	Garlic (5.5–7.5)
	Eggplant (5.5–6.5)	Brussels sprouts (6.0–7.5)	Okra (6.0–7.5)	Parsley (5.0–7.0)
	Garlic (5.5–7.5)	Cabbage (6.0–7.5)	Onion (6.0–7.0)	Parsnip (5.5–7.5)
	Melon (5.5–6.5)	Cantaloupe (6.0–7.5)	Oregano (6.0–7.0)	Peanut (5.0–6.5)
	Parsley (5.0–7.0)	Cauliflower (6.0–7.5)	Pak choi (6.5–7.0)	Pepper (5.5–7.0)
	Pepper (5.5–7.0)	Celery (6.0–7.0)	Parsnip (5.5–7.5)	Rutabaga (5.5–7.0)
	Pumpkin (6.0–6.5)	Chinese cabbage (6.0–7.5)	Pea (6.0–7.5)	Squash, winter (5.5–7.0)
	Radicchio (6.0–6.7)	Celeriac (6.0–7.0)	Radicchio (6.0–6.7)	Tomato (5.5–7.5)
	Radish (6.0–7.0)	Celery (6.0–7.0)	Radish (6.0–7.0)	Turnip (5.5–7.0)

Acidophile crops (pH from 4 to 5.5)	Slightly acidophile crops (tolerate pH from 5.5 to 6.5)	Moderately alkalophile crops (tolerate pH from 6.0 to 7.0 or greater)		Crops of great tolerance: (tolerate a wide range of soil acidity or alkalinity, from about 5.0 to 7.0)
	Rhubarb (5.5–7.0)	Chinese cabbage (6.0–7.5)	Rhubarb (6.5–7.0)	
	Sorrel (5.5–6.0)	Chive (6.0–7.0)	Sage (6.0–6.7)	
	Squash, winter (5.5–7.0)	Cilantro (6.0–6.7)	Salsify (6.0–7.5)	
	Sweet potato (5.5–6.0)	Claytonia (6.5–7.0)	Spinach (6.0–7.5)	
	Tomato (5.5–7.5)	Collard (6.5–7.5)	Squash, summer (6.0–7.0)	
	Turnip (5.5–7.0)	Cress (6.0–7.0)	Sunflower (6.0–7.5)	
		Endive/escarole (6.0–7.0)	Swiss chard (6.0–7.5)	
		Fennel (6.0–6.7)	Tarragon (6.0–7.5)	
		Gourd (6.5–7.5)	Tomatillo (6.7–7.3)	
		Horseradish (6.0–7.0)	Watermelon (6.0–7.0)	

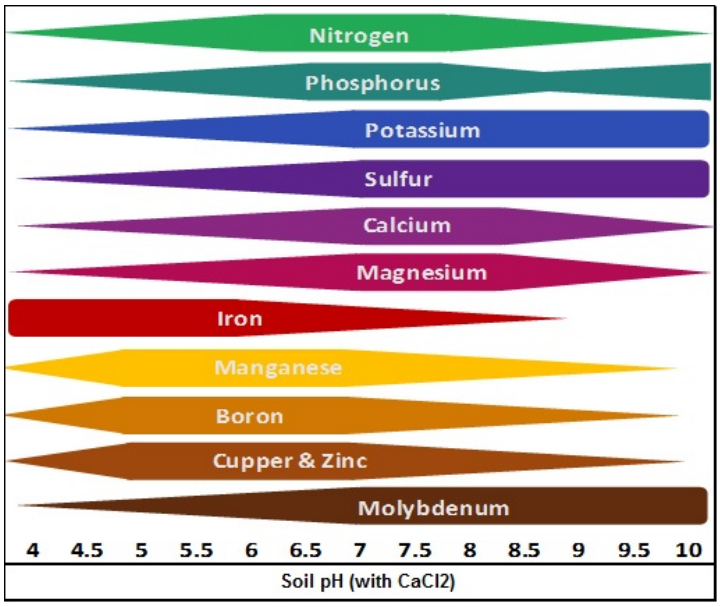

Figure 2.1.12.1 Solubility of plant nutrients as a function of soil pH($CaCl_2$)

Principle

Soil pH is the activity of hydrogen ions (H^+). It is a measure of the acidity/alkalinity of a soil solution on a scale from 0 to 14 (Fig. 2.1.12.2). Acidic solutions have a pH below 7, while basic or alkaline solutions have a pH above 7. Instrumental methods are specified for routine pH determination (ISO 10390:2005): a glass electrode in a 1:5 (volume) suspension of soil in water (pH in H_2O), in 1 mol/L potassium chloride solution (pH in KCl) or in 0.01 mol/L calcium-chloride solution (pH in $CaCl_2$).

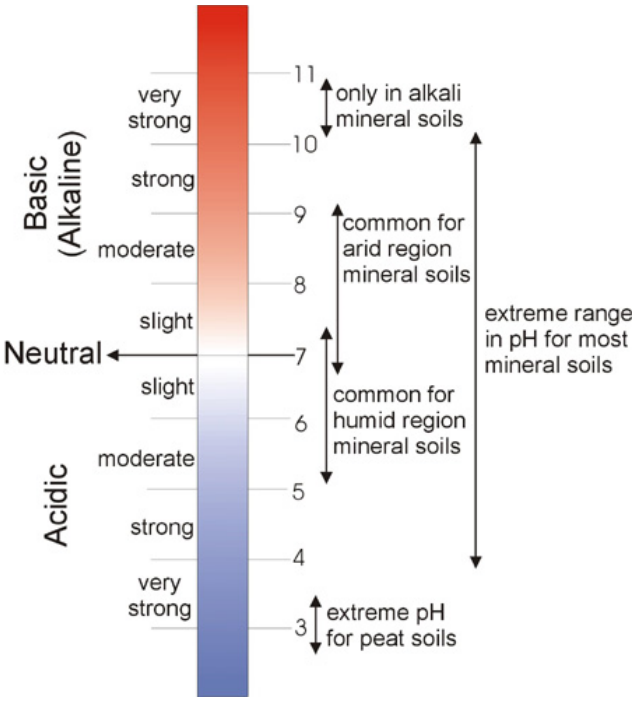

Figure 2.1.12.2 Classification of soils by pH

Reagents

- Distilled H_2O
- a solution of potassium chloride (KCl) in water, c = 1 mol/L
- a solution of calcium chloride ($CaCl_2$) in water, c = 0,01 mol/L

Materials and equipment

- pH meter (based on voltametry)
- pH and reference electrode or combined electrode
- 50 ml beaker
- Scale (0.1g)
- Standard buffers pH4, pH7, pH10

Procedure

Samples should be analysed as soon as possible after being taken.

a. Calibrate the pH meter following the manufacturer's instructions using the buffer depending on the expected values for the soils (Pansu & Gautheyrou, 2006)
b. Prepare 10 g soil (~) and 25 ml solution (FAO, 2006) OR a 1:5 soil: water suspension with 10 g air-dred soil (<2mm) weighed into a bottle and with 50 mL solution added
c. Cover and continuously stir the suspension for 5 min
d. Mechanically shake it for 1 hour at 15 rpm

e. Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension, or filter or centrifuge
f. Immerse the electrode just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole
g. Insert the electrodes into the sample solution. The pH value can be read when the pH value does not change

Remarks

- Field pH measurement should not be a substitute for laboratory determination, but correlated with laboratory analyses where possible.
- A common method for increasing soil pH is to lime soils with calcium carbonate, calcium oxide (CaO), calcium hydroxide ($Ca[OH]_2$) or calcium containing by-products such as sugar-beet lime. The liming material reacts with carbon dioxide and water in the soil to yield bicarbonate (HCO_{3^-}) and hydroxide (OH), which take H+ and aluminium (acid-forming cations) out of solution, thereby raising the soil pH.

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13. Electrical conductivity

Dénes Lóczy and József Dezső

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

The electrical conductivity (EC) of soils correlates with soil properties which influence crop yields (Anderson-Cook et al., 2002), since it is directly related to the content of salts. High concentrations of salts in the soil solution can affect plants in multiple ways: specific toxicity can be due to the abundance of a particular ion (e.g. sodium). Excessive osmotic pressure around the roots prevents an efficient water absorption by the plant. Some crops are more susceptible to salinity than others. Each species has an electrical conductivity threshold, beyond which a reduction in yield must be taken into account. Electrical conductivity measurements are among the most frequently used tools in precision agriculture research with the purposes of describing soil properties and human activities which influence the crop yield (Corwin & Lesch, 2005).

Principle

The measurement of electrical conductivity is based on the ability of a material to transmit (conduct) an electrical current and is commonly expressed in units of milliSiemens per metre [mS/m]. The EC or specific conductance is the reciprocal of electrical resistivity (Ohm, symbol: the Greek letter omega), Ω^{-1}. Its SI unit is Siemens metre^{-1}.

Reagents

- 0.01M potassium chloride reference solution

Materials and equipment

- EC meter
- EC electrode
- 50 mL beaker
- Automatic shaker

Procedure

a. Calibrate the conductivity meter according to the manufacturer's instructions using the KCl reference solution to obtain the cell constant
b. Prepare a 1:5 soil:water suspension by weighing 10 g air-dried soil into a beaker
c. Add 50 mL deionised water
d. Shake at 15 rpm for 1 hour to dissolve soluble salts
e. Rinse the conductivity cell with the soil suspension

Calculation/Evaluation

The electrical conductivity values can be evaluated for soil salinity (Table 2.1.13.1).

Table 2.1.13.1 Salinity classes of soils based on electrical conductivity (Campbell, 2017)

USDA Class	Conductivity Range dS/m	Salt in Soil g/100g	Osmotic potential kPa	Crop Salt Tolerance	Example Crop
A	0–2	0–0.13	0 to -70	Sensitive	Bean
B	2–4	0.13–0.26	-70 to -140	Moderately Sensitive	Corn
C	4–8	0.26–0.51	-140 to -280	Moderately Tolerant	Wheat
D	8–16	0.51–1.02	-280 to -560	Tolerant	Barley

Remark

- There is no clear relationship between electrical conductivity (1:5 soil:water) and total soluble salts due to the different ionic conductivities of the various salts and the influence of the soil particles

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14. Aggregate stability and size distribution

Johan Six and Roman Hüppi

Department of Environmental Science, Institute of Agricultural Sciences, ETH Zurich, Zurich, Switzerland

Importance and applications

This method provides information about aggregate stability and size distribution. It can be used as an indicator for soil structure and how soil structure changes under different management and land use changes. It has also allowed detailed study into how soil organic matter is transformed and stabilised under different management regimes (Six et al., 1998). Lastly, it has been used to identify and study the dynamics of soil microenvironments and their hosted microbial community.

Principle

This method analyses the aggregate stability and size distribution based on a wet sieving method of air-dried soil. (Elliott et al., 1986). The wet sieving of air-dried soil induces the process of slaking (i.e. break-up of non-stable structures in the soil due to a build-up of air pressure within pores upon wetting) and thereby isolates only stable aggregates. In contrast, when the air-dried soil is rewetted, then the process of slaking is minimised and hence less stable aggregates are isolated. In practical terms, this means that field-moist soil is first gently broken up to pass an 8-mm sieve and then air-dried. Subsequently, two pre-treatments are applied before wet sieving: (i) air-dried soil is rapidly immersed in water (slaked treatment) and (ii) air-dried soil is capillary-rewetted before immersion in water (rewetted treatment). For capillary rewetting, air-dried soil is placed on filter paper that is slowly moistened until a water content of 1.05 times field capacity is reached (Six et al., 2000a). Three sieve sizes (2 mm, 0.250 mm, and 0.053 mm) are used to separate the soil into four different aggregate size classes.

Reagents

- No reagents are needed, but deionised water should be used if available

Materials and equipment

- 8 mm sieve
- Two white basins with diameter of 50 cm and height of 8 cm
- 2000 µm, 250 µm, 53 µm sieves with diameter of 30 cm
- Aluminium pans (large and small) for drying of samples
- Air-forced drying oven (60°C)
- Spatula and brush
- Rinsing bottle
- Balance (2 significant digits)

Optional:

- Erlenmeyer flask with tube and pipette tip
- Vacuum pump
- Convection drying oven (105°C)

Procedure

Part A: Wet-sieving of whole soil:

a. Take 80 g (or between 50 and 100 g) subsample from air-dried or rewetted whole soil (weigh on digital balance and record weight; two significant numbers)
b. Fill up white basin (30 cm diameter; 8 cm deep) with water until water level is approximately 1 cm above 2000 µm sieve-mesh
c. Spray soil evenly out on sieve and wait for 5 minutes (to allow the slaking process)
d. After the 5 minutes, sieve the soil for two minutes by moving the sieve up and down (approx. 3 cm amplitude) 50 times with a slight angle to ensure that water and small particles pass through the mesh
e. Depending on the soil, carry out steps 5–10 or instead steps 11–14. Put the sieve down and rinse off the insides of the sieve with water in order to have all particles in suspension
f. Let the sieve stand for 30 sec in order to let small particles settle
g. Aspirate off the floating litter into the first flask attached to the vacuum line
h. When all floating material is aspirated, empty the flask into the waste basket
i. Take the sieve out of the water and rinse off the sides plus the bottom of the sieve with water in order to have all particles in suspension
j. Put the sieve with aggregates into the 105°C convection oven (for 30 min)
k. Take the sieve out of the water and rinse off the sides plus the bottom of the sieve with water in order to have all particles in suspension
l. Backwash > 2000 µm aggregates (i.e. large macroaggregates) into a pre-weighed small drying pan with sufficient water.
m. Decant off the floating litter into the waste bucket.
n. Put the drying pan with the large macroaggregates into the 60°C forced air oven (overnight).
o. Pour the water and particles that went through the 2000 µm sieve remaining in the white basin onto a 250 µm sieve, which is held above the second white basin, and repeat the sieving procedure (in 2 minutes the sieve is moved up and down (approx. 3 cm amplitude) 50 times with a slight angle to ensure that water and small particles pass through the mesh).
p. Take the sieve out of the water and rinse off the sides plus the bottom of the sieve with water in order to have all particles in suspension.
q. Backwash 250-2000 µm aggregates (i.e. small macroaggregates) into a pre-weighed small drying pan.
r. Put the drying pan with the small macroaggregates into the 60°C forced air oven (overnight).
s. Pour the water and particles that went through the 250 µm sieve remaining in the white basin onto a 53 µm sieve, which is held above the second white basin, and repeat the sieving procedure (in 2 minutes move the sieve up and down (approx. 3 cm amplitude) 50 times with a slight angle to ensure that water and small particles pass through the mesh).
t. Take the sieve out of the water and rinse off the sides plus the bottom of the sieve with water in order to have all particles in suspension.
u. Backwash 53–250 µm aggregates (i.e. microaggregates) into a pre-weighed small drying pan.
v. Put the small drying pan with microaggregates into the 105°C forced air oven (overnight).
w. Pour the water + < 53 µm particles (i.e. silt + clay) remaining in the white basin into a pre-weighed large drying pan.
x. Put the large drying pan with silt + clay particles into the 105°C forced air oven (overnight)

y. *If steps 5-10 were chosen:* Take the > 2000 µm sieve out of the convection oven and transfer the large macroaggregates to the pre-weighed small drying pan (do not use any water in this step, just lightly brush aggregates off the sieve into the pre-weighed small drying pan
z. The following day weigh all fractions

Calculations

The results are generally expressed as bar graphs showing the proportions of the different aggregate size classes or are expressed as the mean weight diameter (MWD):

$$MWD = \Sigma A_i * P_i \qquad \text{(Eq. 2.1.14.1)}$$

where

A_i is the mean size of the aggregate size class,

P_i is the proportion of the respective aggregate size class.

Remarks

- If soils with different textures are compared, a sand correction should be performed (see Six et al., 2000b)
- When both the air-dried and rewetted soils are fractionated into different aggregate size classes and sand-corrected, the Normalised Stability Index (NSI) can be calculated, according to Six et al., 2000b. The NSI is a preferable indicator for soil stability for soils with different textures

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15. Soil structure

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

Structure is not only an important property for soil classification but an indicator of soil conditions. Soil structure strongly influences soil hydraulic and solute transport processes, which can be significantly improved or deteriorated through management practices (Bronick & Lal, 2005). Soil structure deteriorates when structural units are deformed. This happens when pressure is applied to a soft soil in wet conditions. Pressure squeezes the soil units together and reduces pore space within the units. A dry soil can withstand pressure without deforming the soil structure. Practices that increase productivity and decrease soil disruption enhance aggregation and structural development. Environmental changes of natural origin also have an impact on soil structure. All these influences should be taken into account when striving for sustainable farming (Six et al., 1999).

Principle

Soil structure is defined by the way individual particles of sand, silt, and clay are assembled. Single particles when assembled appear as larger particles, called *aggregates*. During pedogenesis, clay minerals and Fe- and Al-(hydr)oxides are bound together as microaggregates (i.e. compound soil structures smaller than 250 μm), primarily by physicochemical and chemical interactions involving cementing (e.g. carbonates) and gluing agents (Fe, Mn, and Al compounds) (Totsche et al., 2018). The small aggregates build large fractions, macroaggregates, by combining with organic matter. The structure of a soil refers to both the geometric arrangement of the particles or mineral grains, i.e. soil fabric (Holtz & Kovacs, 1981) as well as the pore spaces that are left between them. The processes of root penetration, wetting and drying cycles, freezing and thawing and animal activity combined with inorganic and organic cementing agents produce soil structure (Snyder & Vázquez, 2005).

There are four major grades of structure (FAO, 2006):

1. *Structureless soils* show no observable aggregation or no definite orderly arrangement of natural lines of weakness.

2. *Weak structure* is poorly formed from indistinct aggregates that can barely be observed in place. When removed from the profile, the soil material breaks down into a mixture of very few entire aggregates, many broken aggregates and much unaggregated material.

3. *Moderate structure* is well formed from distinct aggregates that are moderately durable and evident but not distinct in undisturbed soil. When removed from the profile, the soil material breaks down into a mixture of many distinct entire aggregates, some broken aggregates and little unaggregated material.

4. *Strong structure* is well formed from distinct aggregates that are durable and quite evident in undisturbed soil. When removed from the profile, the soil material consists very largely of entire aggregates and includes few broken ones and little or no non-aggregated material.

The type of structure describes the form or shape of individual aggregates (or the lack of structure):

1. *Structureless categories* (Fig. 2.1.15.1): no aggregation when the soil is dry. *Massive structure* (coherent): where the entire soil horizon appears cemented in one great mass or *single-grain structure* (non-coherent) where the individual soil particles show no tendency to cling together, such as pure sand.

Figure 2.1.15.1 Structureless soils. a. single grain; b. massive

2. *Granular and crumb structures* (Fig. 2.1.15.2) are individual particles of sand, silt and clay grouped together in small, nearly spherical grains. They are commonly found in the A horizon of the soil profile. Both granular and crumb structures have rounded surfaces, but crumb structures are larger.

Figure 2.1.15.2 Granular and crumb-structured soils

3. *Blocky and subangular structures* (Fig. 2.1.15.3) cling together in angular clumps with sharp edges. Blocky structures are cubes with flattened surfaces, sharp edges, while subangular blocky structures are more rounded.

Figure 2.1.15.3 Coarse (Ø 30-50 mm) angular blocky soils

4. *Prismatic and columnar structures* (Fig. 2.1.15.4) are soil particles separated into vertical columns or pillars by miniature, but definite, vertical cracks. Prismatic aggregates are rectangular, elongated with a flattened top, while in columnar structure they have a rounded top.

Figure 2.1.15.4 Prismatic and columnar soils

5. *Platy and lenticular structure* (Fig. 2.1.15.5) is made up of soil particles aggregated into thin plates or sheets piled horizontally on one another. Plates often overlap to a large extent impairing water percolation.

Figure 2.1.15.5 Platy and lenticular soils

Reagents

- None

Materials and equipment

- Shovel or soil core sampler
- Measuring tape
- Plates with photographs for visual comparison

Procedure

a. Dig a soil pit and prepare the soil profile
b. Take an undisturbed sample using a shovel or soil core sampler
c. Carefully tease the soil apart along lines of natural weakness and breaking the soil into structural units
d. Measure the size and describe the shape of structural units
e. Determine soil strength by applying pressure to a 3 cm cube of soil using your forefinger and thumb (Environment Agency, 2010)

Calculations/Evaluation

Table 2.1.15.1 Occurrence of classes and types of soil aggregates

Structure	Appearance	Size of individual aggregates	Soil type examples
Massive and single grain structureless	all horizons	x	Sandy soils, Fluvisols
Granular and crumb	A	Fine (< 2 mm)	Phaeozem, Chernozem
		Medium (2-5 mm)	
		Coarse (> 5 mm)	
Blocky and subangular	B	Fine (< 10 mm)	Brown forest soils
		Medium (10-50 mm)	
		Coarse (> 50 mm)	
Prismatic and columnar	B	Fine (< 20 mm)	Umbrisols, Vertisols
		Medium (20-50 mm)	
		Coarse (> 50 mm)	
Platy	all	Foliated (< 1 mm)	Antroposols, Forest soil (A horizon)
		Platy (1-3 mm)	
		Tabular (3-5 mm)	

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16. Soil micromorphology

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

Soil micromorphology is defined as 'a method of studying undisturbed soil with the aid of microscope and ultramicroscopic techniques in order to identify the different constituents and to determine the mutual relations, in space and time, as far as the latter is possible' (Encyclopaedia of Soil Science, 2008). Soil-micromorphology covers the description, measurement and interpretation of pedofeatures at microscopic level (Bullock et al., 1985). The micromorphological features well reflect short-term changes during field experiments. The evaluation of organic components is important for the study of the impacts of low-input management practices.

Principle

Micropedofeatures classification and evaluation were developed by Kubiena (1938), Brewer (1976), Bullock et al. (1985) and FitzPatrick (1993). The basic components are **mineral and organic components** as the simplest fabric units of the soil. At the microscopic scale, soils consist of a fine-grained **soil matrix (S-matrix)** and the following pedological features related to soil-forming processes (Brewer, 1976):

- *plasma:* mainly fine clay-sized clay mineral particles, organic material of colloid size
- *skeleton grains*: chiefly silicate sand and silt grains embedded in the plasma
- *soil voids: macropores* (> 1-2 μm diameter, up to several cm) and *matrix pores* (< 1-2 μm diameter) in the soil matrix
- Coarse fragments: Roots and tissue residues
- Organic fine materials: cells and cell residues and amorphous organic materials (mono- and polymorphic, punctuations, organic pigments)

Voids are pores filled with air and water. Simple voids are found between skeletal grains (Fig. 2.1.16.1). Compound voids are located between aggregates and their faces do not accommodate each other (Fig. 2.1.16.2), while complex packing voids are between single grains and aggregates (Fig. 2.1.16.3).

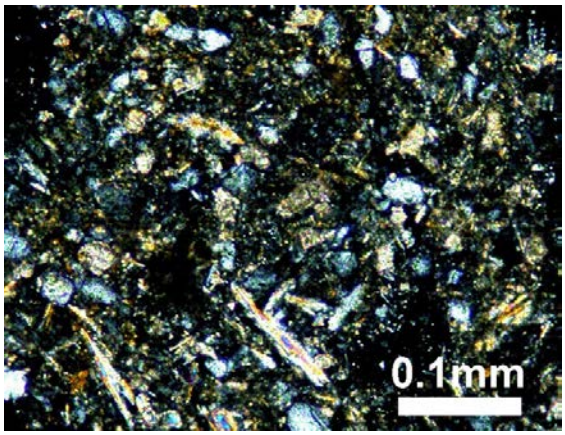

Figure 2.1.16.1 Depleted microstructure with simple pores (voids)

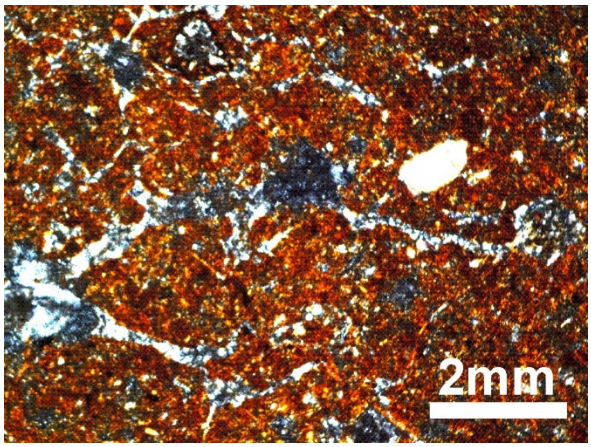

Figure 2.1.16.2 Compound packing voids between aggregates

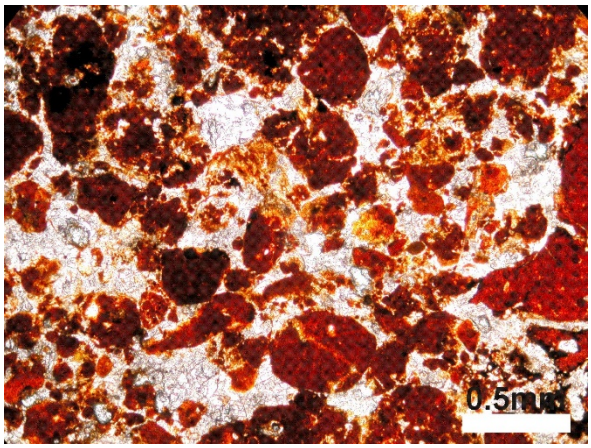

Figure 2.1.16.3 Complex voids in deposited material

Vughs are irregular voids whose origin cannot be attributed to a simple packing of units (Fig. 2.1.16.4). Vesicles are independent, separate features with spherical or elliptical shapes and smooth walls. Channels are tubular forms developed by roots. Chambers are of spherical shape, partly or totally connected with pores or vughs (Fig. 2.1.16.5). Planes are fissures, frequently due to soil desiccation (Fig. 2.1.16.6).

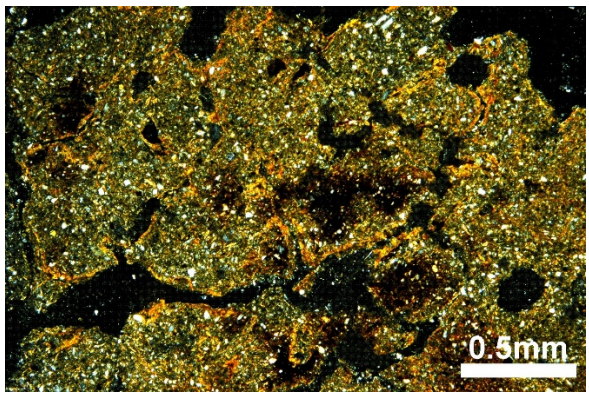

Figure 2.1.16.4 Vughs with birefrigrent clay coatings

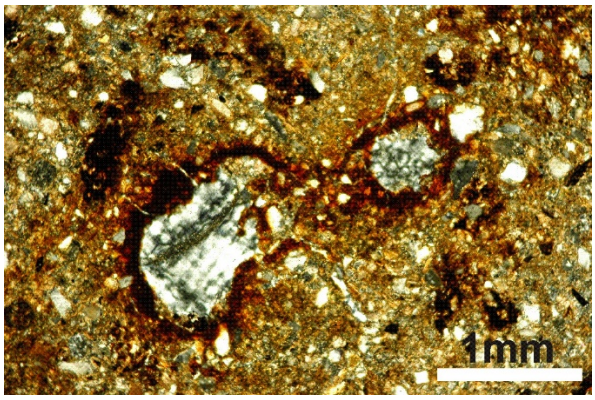

Figure 2.1.16.5 Chambers with coatings

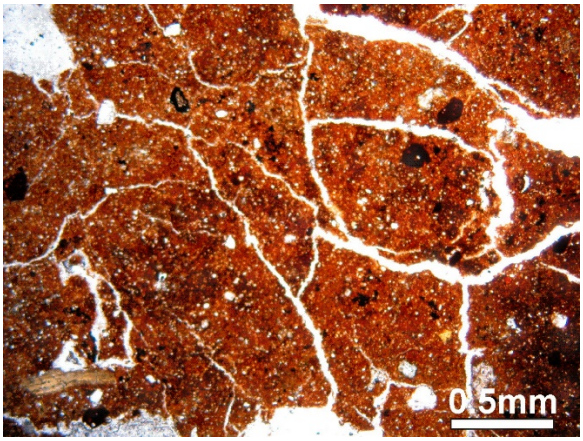

Figure 2.1.16.6 Fissures between angular blocks

Aggregates include crumbs, which are porous aggregates with a spheroidal shape (Fig. 2.1.16.7); granular, non-porous, semi-spheroidal aggregates (Fig. 2.1.16.8); angular blocks with irregular polyhedral shapes (Fig. 2.1.16.9); prismatic angular blocks (Fig. 2.1.16.10); platy, leaf-shaped aggregates (Fig. 2.1.16.11).

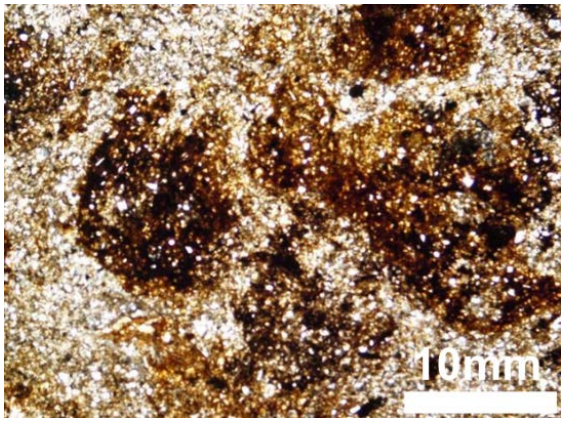

Figure 2.1.16.7 Porous spheroidal crumbs

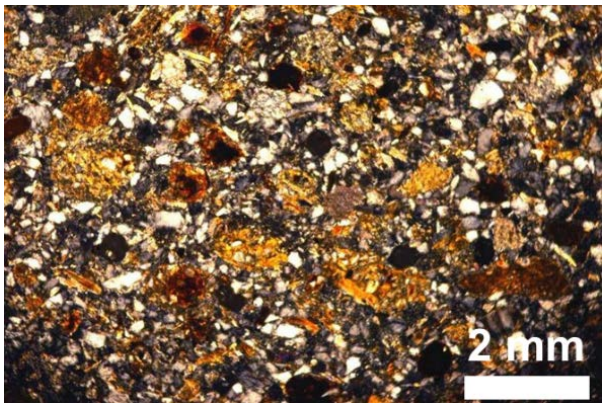

Figure 2.1.16.8 Granular, semi-spheroidal crumbs

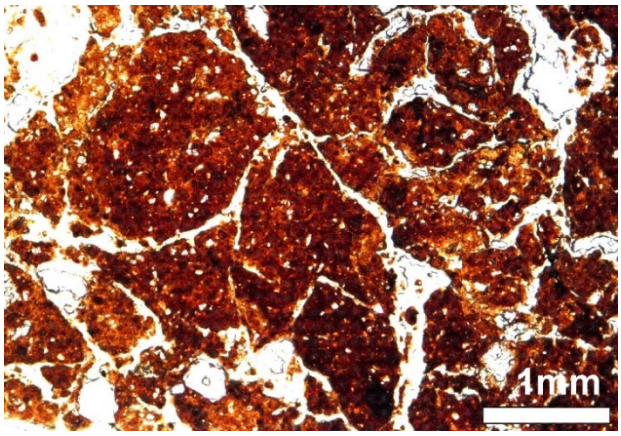

Figure 2.1.16.9 Angular blocks

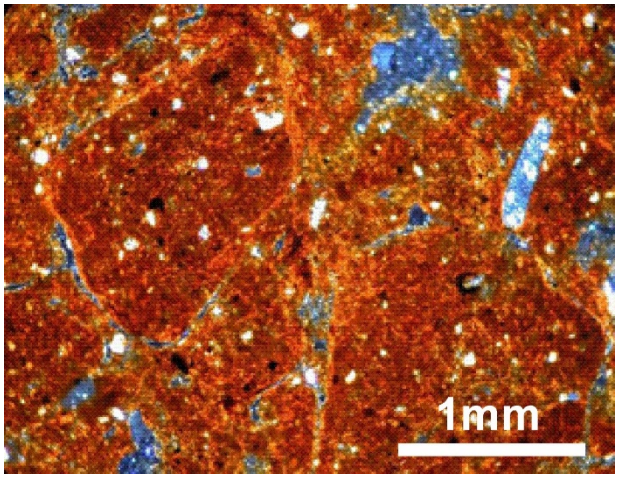

Figure 2.1.16.10 Prismatic angular blocks with birefringent coatings

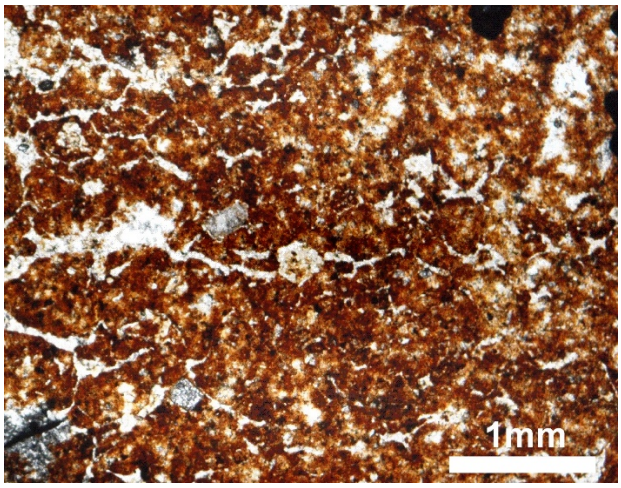

Figure 2.1.16.11 Aggregates in platy structure

The morphology of pedofeatures (related to groundmass) includes

- *Coatings*: defined by their composition and by the surface of their coats (clay, carbonates, gypsum, Fe, Mn and organic compouds; they are not impregnations) (Fig. 2.1.16.12)

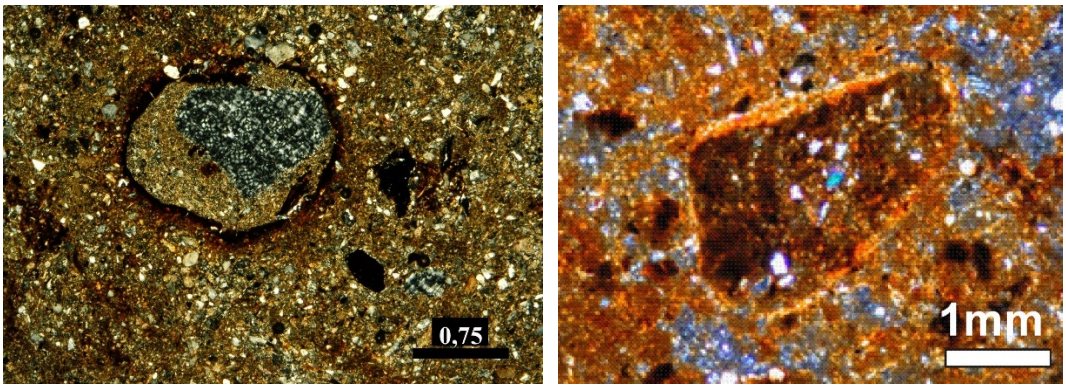

Figure 2.1.16.12 Fe- clayey coatings

- *Hypo-coatings* occur in the matrix, adjacent to natural surfaces (carbonate, Fe, Mn, Fe/Mn compouds; can be of impregnative, depletion or fabric type) (Fig. 2.1.16.13)

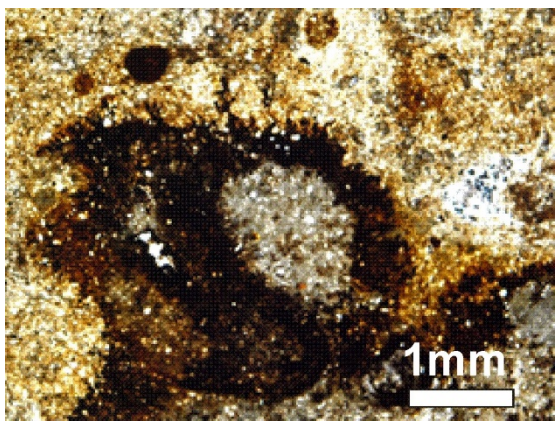

Figure 2.1.16.13 Mn-dendrital hypocoatings around crystallic infilling

- *Infillings* are voids (partially) filled with soil or some fraction (fine material, clay, gypsum, carbonates); totally filled (Fig. 2.1.16.14); continuous infilling with some empty spaces; infilling without continuity, consisting of grains, aggregates, crystals or excrements regularly distributed throughout the entire void (Fig. 2.1.16.15);

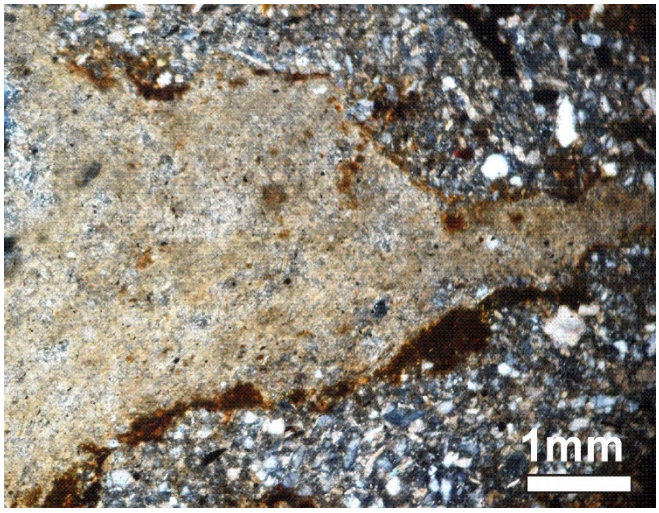

Figure 2.1.16.14 Irregular void with Fe coating filled by carbonate

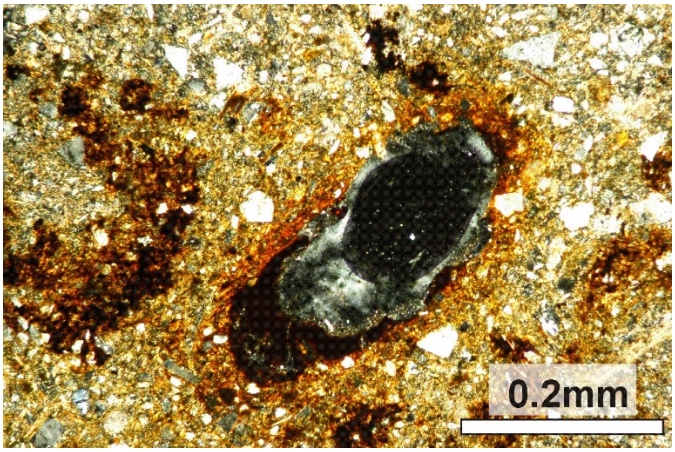

Figure 2.1.16.15 Partly degraded Fe-infilling accumulated in the lower part of channel

Reagents

- Resin
- Acetone
- Oil
- Grinding powder (0.125, 0.050, 0.010, 0.002 mm)
- Diamant paste (< 0.001 mm)

Materials and equipment

- Vacuum chamber
- Grinding machine

- Polishing machine

Procedure

a. Take the undisturbed sample and put it into boxes with double lids to avoid disturbance
b. Use of synthetic resins for improved impregnation
c. Apply acetone as a solvent of the resins to remove water from the samples (reducing shrinkage)
d. Add acetone to the resin to increase viscosity (the amount depends on the density, structure, and composition of the material
e. Use accelerator, hardener, depending on the official instructions for the use of the resin
f. Allow samples to be saturated with resin by capillary rise
g. Place samples into the vacuum chamber, slowly bring to 12 to 28 mercury vacuum to eliminate air bubbles; leave samples under pressure for an initial 24-hour period
h. Add resin and repeat this process twice or three times
i. Final curing in the oven at 50°C for a 24 to 48-hour period
j. Cut the impregnated solid soil blocks into 4-5 mm thin slices
k. Fix the slices on the glass (6 x 4, 8 x 6, 12 x 8 cm respectively)
l. Put the material in the grinding machine and grind to 0.1–0.05 mm thickness
m. Hand-finish using 0.01–0.002 mm grinding-powder and diamant paste,
n. Finish the polishing by the finest polish-powder at 20-50 µm (depending on the subject)

Calculations/evaluation

The thin section is usually described for

- *fabric*: spatial arrangement of material which, formed by particles, constituents
- *colour:* recorded by plane polarised light (PPL), crossed polarised light (XPL), oblique incident light (OIL) settings at different magnification, and/or computerised image analysis
- *grain size:* measured by micrometer. The ‘coarse’ and ‘fine’ limit, calculating or estimating coarse:fine (C:F) ratio is not a fixed value, it depends on the investigated material and aim; 5–15 micrometer (using optical microscope)
- *composition*: single and/or compound mineral grains, fragments; organic materials, residues; inorganic residues of biological origin; human articrafts
- *abundance:* relative percentage of particles
- *shape:* as circularity in two dimensions
- *roundness:* evaluating two-dimensional silhouettes shape (angular, subangular, subrounded or rounded)
- *sorting:* degree of variability (well-sorted, moderately sorted, unsorted bimodal [in the case of two component groups])
- *feature and pattern*: predicted change in the size of microaggregates, abudance of opaque organic materials, distribution of roots, fillings or depletions of channels, cracks, compaction or lack of bioturbation, etc.

Remarks

- The interpretation of features and patterns is based on experience combined with aims. The recognition of individual features is a complex task. Experts have to focus on soil micromorphological features which are able to alter within a short period (3 years) of research or will change with treatments

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17. Runoff coefficient and infiltration rate

Thomas Iserloh, Manuel Seeger

Department of Physical Geography, Trier University, Campus II – Behringstr. 21,

54296 Trier, Germany

Importance and applications

The occurrence of extreme rainfall events severely affects society and can have a significant economic impact (de Lima et al., 2013; Kovats et al., 2014). High runoff rates may lead to increased soil erosion, especially when runoff concentration occurs and thus, linear erosion increases. On the other hand, reduced infiltration rates may lead to reduced water retention in the soil; they may also be indicators for crusting and compaction of the soil.

Principle

The runoff coefficient (%) interrelates the amount of runoff to the amount of precipitation received. It has a higher value for areas with low infiltration and high runoff and lower for areas with high infiltration rates. The infiltration rate (mm h^{-1}) is the amount of water entering the soil within a certain time interval. As runoff and infiltration are highly variable at temporal and spatial scales, in-situ measurements are difficult.

By means of rainfall simulation experiments, a calibrated uniform and reproducible rainfall with a defined intensity is sprayed on a delimited plot (Iserloh et al., 2013). The total runoff produced during a defined duration is collected at plot outlet. Runoff coefficient and infiltration rate are calculated afterwards.

Reagents

- Water

Materials and equipment

A small portable rainfall simulator (described in detail by Iserloh et al., (2012) equipped with:

- Plot
- Framework
- Nozzle holder with nozzle
- Rubber tarpaulin as cover against wind influence
- Water barrel
- A rod to mount flow control
- Flow control
- Bilge pump with battery and battery charger
- Hose connecting pump and flow control,
- Hose connecting flow rate meter and nozzle

Tools:

- Geometer (yardstick)
- Rubber hammer to drive in the plot
- Small shovel
- Screwdrivers (normal and phillips)
- Level (to align the irrigation setup horizontally and vertically)

- Pipe wrench

Other requirements:

- Clinometer including a compass (to measure plot inclination and orientation)
- Camera (for documentation)
- Plumb bob (on a string)
- Water canister
- Wide-neck plastic bottles (250, 500 ml)
- Graduated beakers
- Board with chalk
- Data recording sheets
- Silicon band
- Stopwatch

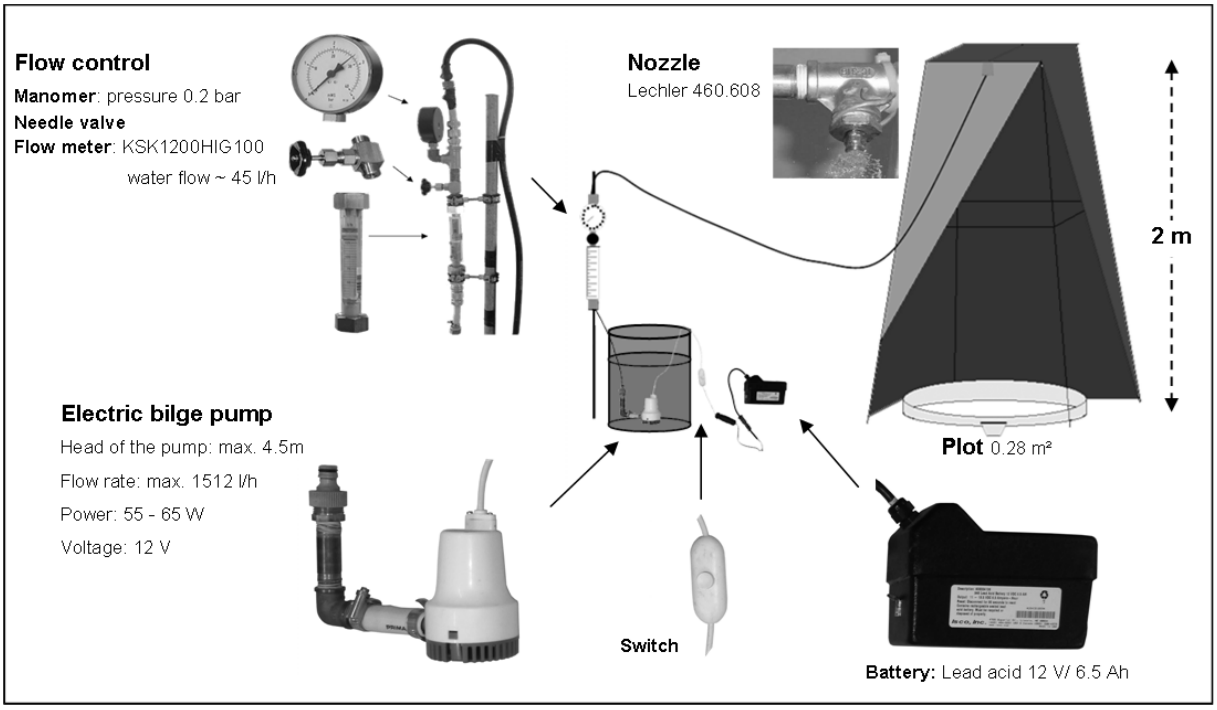

Figure 2.1.17.1 Small portable rainfall simulator (Iserloh et al., 2012).

Procedure

a. Place the plot on the soil and ensure that the runoff shield is oriented to point downhill
b. Carefully drive the plot into the soil with a rubber hammer
c. In order to position the collection containers, dig a small hole underneath the runoff shield
d. Map and photograph the soil surface
e. Place the calibration plate on the plot (thus, the plot is protected)
f. Before installing the framework, check hoses, connectors, flow meter and especially the nozzle for dirt (and clean if necessary). Check battery load
g. Set up the rainfall simulator:
h. Install the framework with wind protection

i. Orient and fix the simulator: Lock the nozzle in place at the correct position over the plot surface by using the plumb lead and arrange the nozzle holder vertically using a level
j. Place the flow meter on the rod close to the plot
k. Place the water barrel close to the irrigation setup and fill with water (at least 50 L)
l. Connect the bilge pump to the flux meter and this to the nozzle with the hoses
m. Place the pump into the water barrel and connect to the battery
n. Turn on pump and regulate to desired flow
o. Calibrate rainfall intensity by irrigating for 2.5 minutes on a calibration plate covering the whole plot and collect the runoff in a calibration vessel

Experimental procedure:

p. Start the measurement immediately after successful calibration without interruption by removing the calibration plate from the plot
q. After runoff starts, collect runoff water in plastic bottles of 250 ml and 500 ml capacity (depending on the amount of runoff, several bottles will be needed)
r. Record runoff start and time for every change of bottle

Calculations

a. Calibration of rainfall intensity on the plot (mm h^{-1}):

$$I\ [mm\ h^{-1}] = \frac{Water\ volume\ [L]}{Time\ [h] \times Plot\ area\ [m^2]} \quad \text{(Eq. 2.1.17.1)}$$

Runoff coefficient [%]: $RC\ [\%] = \frac{Runoff\ [L]}{Precipitation\ [L]} \times 100$ (Eq. 2.1.17.2)

b. Infiltration rate (mm h^{-1}):

$$I\ [mm\ h^{-1}] = Precipitation\ [mm\ h^{-1}] - Runoff\ [mm\ h^{-1}] \quad \text{(Eq. 2.1.17.3)}$$

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18. Sediment load and concentration

Thomas Iserloh, Manuel Seeger

Department of Physical Geography, Trier University, Campus II – Behringstr. 21,

54296 Trier, Germany

Importance and applications

Soil erosion is now being recognised as a severe threat to socio-ecological security and stability. The manifold issues concerning soil health involve aspects as fundamental as food security, resilience to climate change and geosocial stability (Marzen et al., 2017). Soil erosion by water is generally expressed by sediment output (total sediment load and sediment concentration).

Principle

Gerlach troughs are built, installed and utilised as sediment collectors (Gerlach, 1967). Amounts of soil loss, surface flow and sediment concentration are calculated in g, L and g L^{-1}, respectively. Open soil erosion plots give information about the soil (g) and water losses (L), but the contributing area is not defined and may be variable. Consequently, soil erosion or overland flow are shown in g m^{-1} and L m^{-1} of slope width, respectively. Sediment output of a definable field section is measured under real agricultural conditions. The collected material will provide basic data on the transported grain sizes and nutrients of the particular field (Schmidt, 1979).

Reagents

- Water

Materials and equipment

Gerlach troughs are located at the bottom of each crop field studied. They are equipped with a slanted front edge to prevent scouring or undercutting of the trough. Additionally, they can be connected to collecting tanks to be prepared for extreme rainfall events, which can exceed the total storage capacity of the collector.

Material:

- Gerlach trough. Custom construction from galvanised sheet metal (1mm wall thickness). The dimensions are shown in Figure 2.1.18.1.

Equipment:

- Trowel
- Windscreen cleaner
- Scraper
- Brush
- Wash bottle
- Buckets with lids
- Funnels and filters
- Drying cabinet
- Precision scale

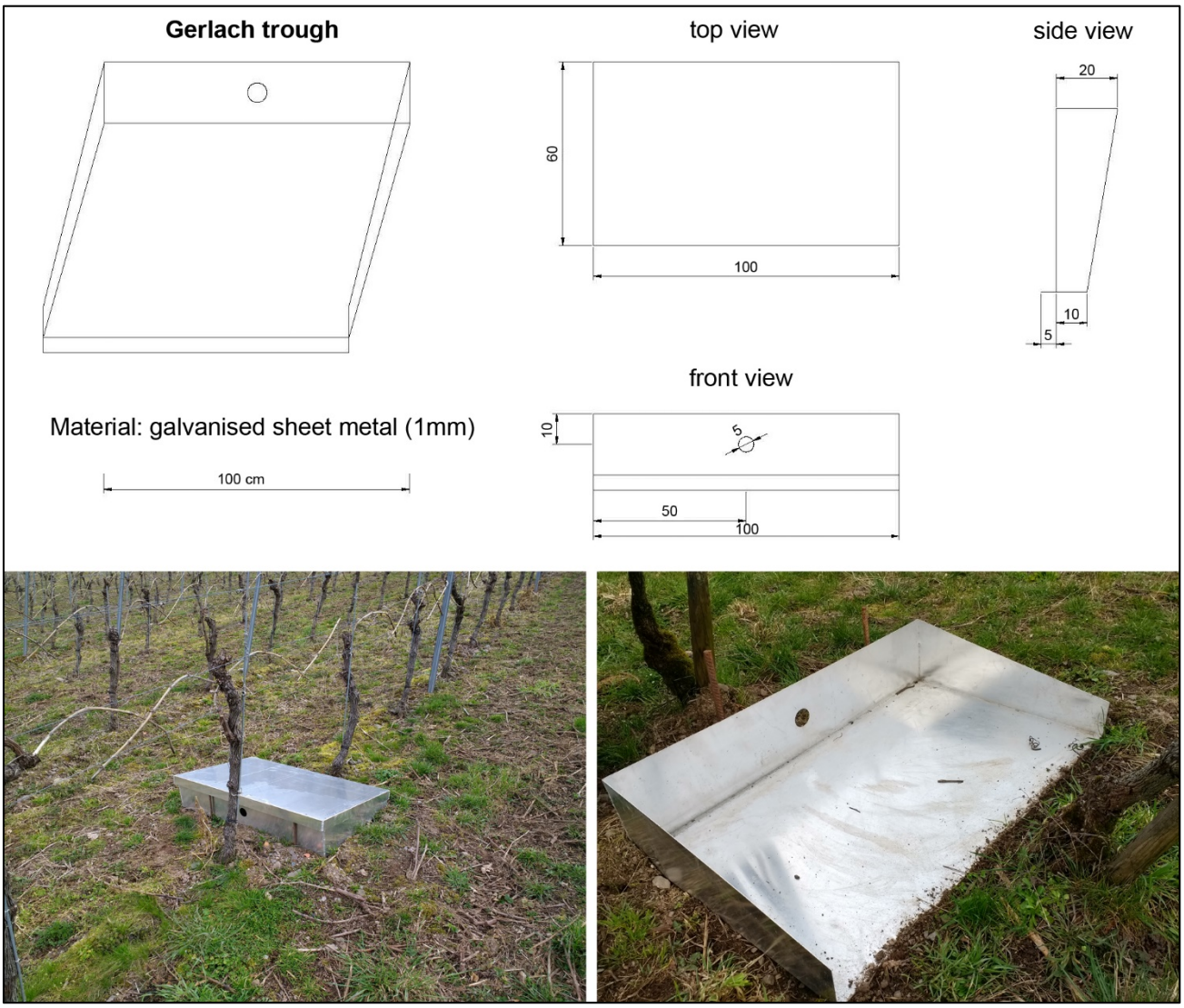

Figure 2.1.18.1. Gerlach trough

Procedure

a. Install the Gerlach troughs at the lower field edges. The field must be uniformly inclined, best stretched out and exposed in one direction only.
b. Clearly delimit the catchment area.
c. Ensure a smooth transition between soil surface and Gerlach trough.
d. Empty the Gerlach troughs after every heavy rainfall event or after constant rain, at least once a week.
e. Collect total surface flow and soil loss by means of trowel, windscreen cleaner, scraper, brush and wash bottle until Gerlach trough is completely clean.
f. Fill total surface flow and soil loss and transport them in buckets with lids.
g. Determine total runoff by measuring the amount of water, in measuring cups.
h. Determine total soil loss by filtration of the samples in the laboratory. Weigh the amount of eroded material after filtering and air-drying (to allow for further investigations, e.g. particle size analysis).
i. Calculate sediment concentration by dividing the total soil loss by the total runoff.

Calculations

a. Sediment load (g m^{-1}):

$$SSL\ (g\ m^{-1}) = \frac{Sediment\ load\ (g)}{trough\ width\ (m)} \quad \text{(Eq. 2.1.18.1)}$$

b. Sediment concentration (g L^{-1}):

$$SSC\ (g\ L^{-1}) = \frac{Sediment\ load\ (g)}{Runoff\ (L)} \quad \text{(Eq. 2.1.18.2)}$$

Remarks

- none

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19. Rill and ephemeral gully density, cross-sectional profile

Thomas Iserloh, Manuel Seeger

Department of Physical Geography, Trier University, Campus II – Behringstr. 21

54296 Trier, Germany

Importance and applications

The erosion caused by concentrated flows generates rills (depth: 0.02 m–0.1 m) and ephemeral gullies (depth: 0.1 m–0.8 m) of small dimensions that can reach several tens of metres in length. The development of these rills and ephemeral gullies may increase erosion rates to an order of magnitude higher than erosion caused by non-concentrated surface flows (Cerdan et al., 2002; Merz and Bryan, 1993; Nouwakpo et al., 2016a; Poesen, 1987; Wirtz et al., 2012). Because of their highly erratic appearance and their easy removal by soil management, such erosion processes are difficult to quantify (Casalí et al., 2006; Giménez et al., 2009; Nouwakpo et al., 2016b; Wells et al., 2016).

Principle

Mapping in the field by visual identification and recording on the prepared mapping basis (DVWK, 1996). Characterisation by reference to their size (width and depth) as well as form. Measurement of rill and ephemeral gully length and building up relation between rill and ephemeral gully length and mapped area (rill and ephemeral gully density). Rill and ephemeral gullies may be also mapped by interpretation of high-resolution aerial photographs taken by unmanned aerial vehicle (UAV) (Aber et al., 2010). Cross-sectional profiles of rills and ephemeral gullies are determined after methods from Casalí et al., (2006) and Giménez et al., (2009). Simplified estimation of rill and ephemeral gully volume is possible (see below).

Reagents

- none

Materials and equipment

- Detailed map and (recent) aerial photography.
- Field mapping equipment:
  - Scale and yardstick
  - Compass, inclination measuring device

Alternatively:

- UAV with optical camera
- Computer with SfM (Structure from motion) software (e.g. Agisoft Photoscan, Visual SFM)

Procedure

a. Identify linear erosion features in the field
b. Record on the mapping base with appropriate symbols
c. Classify features by
   - size (rill, ephemeral gully)
   - form (v-shaped, rectangular, u-shaped).

d. Record their
   - length [m]
   - depth and width [m] at representative cross-sections of the feature.

e. Optional: digitise into GIS (geographic information system).

Alternative procedure:

a. Take aerial photography by UAV
   - Overlap 80%, lateral overlap > 60%, if possible also oblique images
   - Ground resolution ~0.1 m
   - Point cloud and DEM (digital elevation model) generation by means of appropriate SfM-software
   - Orthophoto as basis for visual rill and ephemeral gully identification
   - (Volume calculations are until now a subject for scientific development).

Calculations

a. Rill and ephemeral gully density:

b. $$Rill \text{ and ephemeral gully } density \left[\frac{m}{m^2}\right] = \frac{Rill \text{ and ephemeral gully } length\ [m]}{Research\ area\ [m^2]} \quad \text{(Eq. 2.1.19.1)}$$

c. Cross-sectional area:

d. $$V - shaped\ area\ [m^2] = \frac{1}{2} \times depth\ [m] \times width\ [m] \quad \text{(Eq. 2.1.19.2)}$$

$$U - shaped\ area\ [m^2] = depth[m] \times width[m] \times \pi \quad \text{(Eq. 2.1.19.3)}$$

$$Rectangular - shaped\ area\ [m^2] = depth[m] \times width\ [m] \quad \text{(Eq. 2.1.19.4)}$$

e. Estimation of rill and ephemeral gully volume:

$$Volume\ [m^3] = area\ [m^2] \times length\ [m] \quad \text{(Eq. 2.1.19.5)}$$

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20. Gully depth, gully growth rate

Thomas Iserloh, Manuel Seeger

Department of Physical Geography, Trier University, Campus II – Behringstr. 21,

54296 Trier, Germany

Importance and applications

Gullies (depth: > 0.8 m) are three-dimensional erosion forms that may appear in various shapes, sizes and complexities (Poesen et al., 2003). Gully erosion destroys agricultural land and is difficult to quantify because of the size and complexity of the forms as well as the tempo-spatial variability of their development (Castillo & Gómez, 2016). Moreover, gully growth is the result of many different processes, such as headwall retreat, lateral collapses and incision (Marzolff & Ries, 2007).

Principle

For the quantification of gully-erosion processes, detailed aerial photographic monitoring has proved to be the most efficient method (d'Oleire-Oltmanns et al., 2012; Eltner et al., 2016; Marzolff & Poesen, 2009; Ries & Marzolff, 2003; Stöcker et al., 2015; Wang et al., 2016). Growth rates are determined either by measurement of gully volume changes or by measurement of headcut retreat rates (Marzolff et al., 2011).

Reagents

- none

Materials and equipment

- UAV (unmanned aerial vehicle) with optical camera
- Computer with SfM (Structure from motion) software (e.g. Agisoft Photoscan, Visual SFM)

Procedure

a. Gully depth: Take aerial photography by UAV
   - Overlap 80%, lateral overlap > 60%, if possible also oblique images
   - Ground resolution ~0.25 m
   - Point cloud and DEM (digital elevation model) generation by means of appropriate SfM-software
   - Orthophoto as basis for visual delineation of the gully edge.
   - Identification of the uppermost gully headcut point.
   - (volume calculations are until now a subject for scientific development)

b. Gully growth rate: As a simple and common measure of gully development, linear retreat rates reflect the average annual backward migration of gully heads in the upslope direction of the drainage line and thus the increase in gully length (Marzolff et al., 2011).

Calculations

a. Gully depth: Calculation from DEM

b. Gully growth rate:

$$\text{Gully growth rate } \left[\frac{m}{a}\right] = \frac{headcut\ retreat\ [m]}{year\ [a]} \quad \text{(Eq. 2.1.20.1)}$$

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21. Soil water content at field capacity and wilting point

József Dezső[a], Dénes Lóczy[a], Alejandro Pérez-Pastor[b], Abdelmalek Temnani Rajjaf[b], David Pérez Noguera[b]

[a]Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

[b]Technical University of Cartagena (UPCT), Department of Plant Production, Paseo Alfonso XIII, 48, ETSIA, 30203 Cartagena, Murcia, Spain

Importance and applications

Soil moisture conditions are closely linked to pore volume, pore size distribution, capillary rise capacity and the groundwater table. Water tension controls the germination time of seeds. In orchards and for horticultural crops, irrigation design is based on the prediction of the dynamics of soil moisture status. Actual soil moisture content (m m^{-1}% or V V^{-1}%), however, only provides limited information on soil-plant system hydrodynamics. The available water (AW) for plant uptake is closely associated with the soil water budget (WB) (Kirkham, 2014). This is a changeable parameter, which must be investigated within the total range of water capacity. This range covers two characteristic points: the field capacity, and the wilting point.

Principle

Field capacity (FC) corresponds to the upper limit of AW and represents the soil moisture content left behind after the water contained in the macropores is drained by gravity (Assouline & Or, 2014). Wilting point (WP) refers to the water content when the soil becomes dry and plants can no longer take up water.

For the determination of FC, soil samples are dried by raising the air pressure in an extractor with a porous ceramic plate. The pores of the plate are filled with water and prevent high-pressure air from flowing through. Soil moisture will flow around the individual soil particles, through the ceramic plate and an outflow tube until equilibrium is reached (UGT, 2018). FC moisture is represented by the balance moisture with tension of 6–33 kPa, depending on soil texture, structure and organic matter content. Richards and Weaver (1949) found that water content held by soil at a potential of −33 kPa correlate closely with FC (−10 kPa for sandy soils). Permanent WP is commonly approximated as the soil water content at –1500 kPa.

The working range of the vacuum gauge tensiometers is limited (Ψm from 0 to -80 kPa) as opposed to the -200 kPa of the WATERMARK-type resistance sensor. Such values suppose no limitation to scheduling localised irrigation, since they can be useful in the application of deficit irrigation strategies, in order to avoid promoting severe water stress to the crop (Pérez-Pastor et al., 2009). The values of Ψm obtained with tensiometers have been widely used in woody crops (Pérez-Pastor et al., 2016). Kaufmann and Elfving (1972) found a good correlation between the readings of the tensiometers and the leaf water potential before dawn (Ψa). The suction value ψ is typically expressed with pressure units (kPa), pressure head (m of water), or centimetric logarithmic head (pF); at 20°C, 9.8 kPa ≡ 100 cm of water ≡ pF of 2 (Aubertin et al., 2003) (Table 2.1.21.1).

Table 2.1.21.1 Conversion between different suction values

kPa	Bar	H_2Ocm	pF	Meaning
0	0	0	1	Saturated soil
-33	-0.333	-336.50	2.5	Field Capacity (FC)
-1500	-15	-15849.00	4.20	Wilting Point (WP)

The sensors that measure the matric potential are based on the direct measurement of the soil water tension (tensiometers), and indirectly of the electrical resistance of the soil (granular matric sensors) and the dielectric permeability of the soil matrix (porous ceramic disc sensors) (Smith & Mullins, 2001). *In-situ* capillary potential may be measured using a tensiometer consisting of a water-filled porous cup connected to a manometer or pressure transducer or, alternatively, by the scattering of neutrons or absorption of gamma rays from a radioactive source (Vaz et al., 2013). The essential part of the instrument is a pipe with a small volume of water reservoir and the tensiometer or irrometer (above ground) ending in a ceramic tip (Fig. 2.1.21.1). The leaking generates vacuum in the pipe according to soil moisture conditions. This rapid field method is applicable between 0 and 30 kPa.

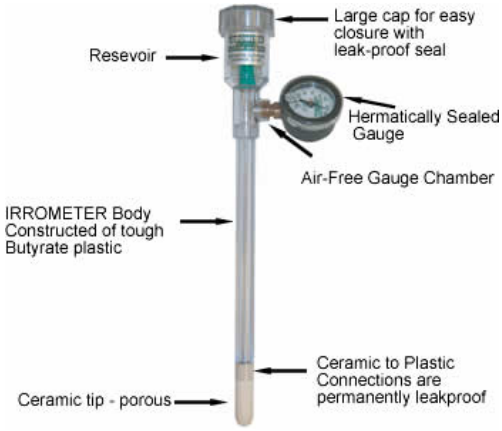

Figure 2.1.21.1 Irrometer (source: Calafrica, 2018)

Reagents

- Distilled water

Materials and equipment

- Auger for undisturbed soil sampling

- *Equipment for determinating actual (in situ) FC:*

  - Auger to prepare the hole for the tensiometer
  - Tensiometer
  - Matric potential sensors (MPS-6, now called Teros 21, METER GROUP)

- *Equipment for determining FC, WP by pressure ceramic plate exactors at different vacuum levels in the laboratory*
  - Pressure control panel equipped with two manometers 0-2 MPa and 0-0.4 MPa
  - Pressure vessel(s)
  - Compressor (220 V/50 Hz); maximum pressure 2.0 MPa,
  - Pressure ceramic plates, 0.1, 0.3, 0.5 and 1.5 kPa standards

Procedure

Determination of actual FC by tensiometer (water-filled porous cup) in the field:

- Prepare a perfect hole in the soil horizon
- Fit the tensiometer into the hole
- Check if fitting is tight between the soil surface and ceramic tip
- Wait 10-30 minutes for the balance between the internal and external part of the ceramic tip (depending on soil textural properties)

Determination of FC and WP by pressure plate exactor at different pressure levels in the laboratory:

- Saturate the undisturbed soil sample with distilled water
- Place the samples on the ceramic plate of 0.1 kPa type.
- Set the compressor to the adequate pressure level at 33 kPa (for FC)
- Measure the soil water content (SWC) of FC when equilibrium is reached
- Change the ceramic plate to 1.5 kPa type
- Set the compressor to the pressure 1500 kPa (for WP)
- Measure the soil water content (SWC) of WP when equilibrium is reached

Calculations

The amount of water held by the root zone of the soil between FC and WP (available for plants):

$$AW = FC - WP$$

Remarks

- When establishing the WRC curve, hysteresis (difference in the rates of saturation and desiccation) may result in up to 20% variation.
- When determining FC and WP it is difficult to know how much time is needed for the wet soil sample to reach the moisture content appropriate at actual pressure (i.e. suction).

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22. Saturated Hydraulic Conductivity

Raúl Zornoza, José A. Acosta, Silvia Martínez, Virginia Sánchez-Navarro, Ángel Faz

Sustainable Use, Management, and Reclamation of Soil and Water Research Group, Department of Agrarian Science and Technology, Universidad Politécnica de Cartagena,

Paseo Alfonso XIII, 48, 30203, Cartagena, Spain

Importance and applications

Saturated hydraulic conductivity measured in the field (K_s) is one of the most important hydrological properties of soil. In agro-ecosystems this property provides information about the internal drainage of soils, highlighting good soil structure or compactness/saturation that may hinder proper water flow in the soil profile. This is important for understanding and characterising the hydrological cycle and the transfer of contaminants transported by water (Lassabatère et al., 2006). This property also informs about possible water logging problems and runoff after intense rainfall events. In irrigated agriculture, the K_s can be used in designing water application rates for drip and sprinkler systems, and thus avoid water-logging and runoff (Mbagwu, 1995).

Principle

The method proposed here to determine K_s was developed by Bagarello et al., (2012). For this, a simple annular ring is inserted at a short depth into the soil, to produce minimal disturbance of the porous medium, and the infiltration time is measured of a few small volumes of water repeatedly applied at the surface of the confined soil. The acronym SBI was suggested by Bagarello et al., (2012) to denote this method, given that it is a 'Simplified method based on a Beerkan Infiltration run'.

Reagents

- Water

Materials and equipment

- A metal ring of internal radius r = 0.075 m (15 cm diameter). Its height can vary, but should be at least 10 cm
- Hammer
- Plastic measuring cylinders (150 mL)
- Plastic beakers (200 mL)
- Timer

Procedure

a. Remove surface vegetation and litter
b. Insert the ring to a depth (d) of about 1 cm into the soil surface with the help of a hammer to avoid lateral loss of the pondered water at the soil surface
c. Pour a known volume of water (0.150 L) into the ring using a cylinder. Immediately when the amount of water is totally infiltrated, measure and record the elapsed infiltration time (s), and pour a second identical amount of water into the ring. Again, record the time (s) needed for the water to infiltrate (cumulative time)
d. Repeat step c. until the difference in infiltration time between five consecutive trials becomes negligible, indicating a practically steady state of infiltration

e. Record the number of water additions (*N*)

Calculations

a. Calculate the experimental water infiltration "*I*" as follows:

$$I \text{ (L m}^{-2} = \text{mm)} = \frac{Poured\ water\ volume\ (L)\ at\ each\ time}{\pi r^2\ (\text{m})} \qquad \text{(Eq. 2.1.22.1)}$$

b. Calculate the experimental cumulative infiltration "*I*" as follows at each cumulative time *t* (s):

$$\text{Cumulative } I \text{ (mm)} = \sum_{t=1}^{N} I \qquad \text{(Eq. 2.1.22.2)}$$

c. Make a plot of the cumulative infiltration, *I,* vs time, *t,* such as that shown in Fig. 2.1.22.1:

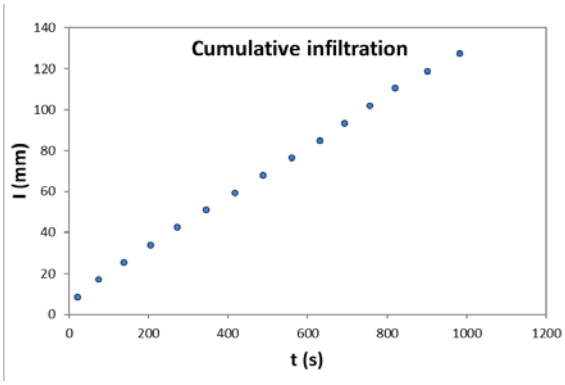

Figure 2.1.22.1. Cumulative infiltration plot

d. Calculate the **infiltration rate**, *IR* (mm s^{-1}) by the slope of the linearised cumulative infiltration curve (Figure 1.22.1), estimated by a linear regression analysis of the (I / $\sqrt{t}$, $\sqrt{t}$) data collected during the steady-state phase of the infiltration run.

e. Calculate K_s as follows:

$$K_s \text{ (mm s}^{-1}) = \frac{IR}{0.467\left(1+\frac{2.92}{r\,\alpha^*}\right)} \qquad \text{(Eq. 2.1.22.3)}$$

where

IR (mm s^{-1}): is the infiltration rate

r (m): is the radius of the ring

α* (mm^{-1}): 0.0262 + 0.0035 x ln(*IR*) (Eq. 2.1.22.4)

Table 1.22.1. Saturated Hydraulic Conductivity classes according to the Natural Resources Conservation Service of the USDA

K_s class	K_s rate (mm s^{-1})
Very rapid	141.14 10^{-3}
Rapid	42.34 10^{-3}–141.14 10^{-3}
Moderately rapid	14.11 10^{-3}–42.34 10^{-3}
Moderate	4.23 10^{-3}–14.11 10^{-3}
Moderately slow	1.41 10^{-3}–4.23 10^{-3}
Slow	0.42 10^{-3}–1.41 10^{-3}
Very slow or impermeable	0.00–0.42 10^{-3}

Remarks

- According to the literature, ***α**** can be estimated on the basis of a general description of soil textural and structural characteristics. However, Bagarello et al., (2012) developed the explained relationship between ***α**** and *IR*, working with 149 infiltration curves collected on Burundian soils. These authors suggested that the infiltration rate contains the necessary information to estimate ***α****.

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23. Actual field soil moisture

Pérez-Pastor, Alejandro[a]*; Temnani Rajjaf, Abdelmalek[a]; Pérez Noguera, David[a]

József Dezső[b] and Dénes Lóczy[b]

[a]Technical University of Cartagena (UPCT), Department of Plant Production, Paseo Alfonso XIII, 48, ETSIA, 30203 Cartagena, Murcia, Spain
*Corresponding author at: Dpto. Producción Vegetal, ETSIA—Universidad Politécnica de Cartagena, Avda. Paseo Alfonso XIII, 48, E-30203 Cartagena, Murcia, Spain. Fax: +34 968 325793. E-mail address: alex.perez-pastor@upct.es.

[b]Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6. Hungary

Importance and applications

The measurement of soil moisture content is fundamental for many studies in agriculture; it measures the amount of water that is retained among the solid particles of the soil and can be expressed as an absolute amount, although it is usually expressed as a fraction of a determined base constant (Jones, 2006). Basically, soil moisture (Soil Water Concentration, SWC) is the water that is held in the spaces between soil particles. Soil moisture is a changeable parameter which governs a whole range of soil processes. It is a major control on the water availability of cultivated crops.

Soil moisture content impacts on many fundamental biophysical processes (Bittelli, 2011): on germination, plant growth, microbial decomposition of the soil organic matter, and nutrient transformations in the root zone. Heat and water transfer at the land–atmosphere interface is also dependent on moisture content. As a major reservoir for water within a catchment, soil moisture directly influences susceptibility to soil erosion and slope stability.

Principle

A wide range of focuses and instrumentation are available for the direct and indirect measurement of the soil moisture content (Smith & Mullins, 2001), of which the most notable are taking soil samples (gravimetric and volumetric methods), neutron probes, Time-Domain Reflectometry (TDR), and Frequency-Domain Reflectometry, (FDR) (Smith & Mullins, 2001). The FDR sensors present several advantages over the other techniques used, amongst which one can highlight: low cost, robustness, that the salt content of the soil does not affect them nor do temperature variations (Paltineanu & Starr, 1997) and that they are easily automated (Martí et al., 2013; Starr & Paltineanu, 1998).
Actual soil moisture content (SWC_a) is often measured *gravimetrically* by drying a soil sample under controlled conditions (Reynolds, 1970a,b,c). For soil moisture monitoring, Time Domain Reflectrometry (TDR) sensors are also used. ***Permittivity*****,** ε (Greek letter epsilon) is the measure of a material's ability to resist an electric field (Davood et al., 2012). By definition, perfect vacuum has a relative permittivity of exactly 1. The difference in permittivity between vacuum and air can often be considered negligible, as κ_{air} = 1.0006. and for water: κ_{water} = 80.

$$K = \frac{\varepsilon}{\varepsilon_0} \qquad \text{(Eq. 2.1.23.1)}$$

The lowest possible permittivity is that of a vacuum. Vacuum permittivity (or electrical constant) is represented by ε_0 and has a value of approximately 8.85×10^{-12} F/m (Faraday/metre). Due to the relative permittivity of materials, an electromagnetic wave travelling through them will experience an increase in the characteristic velocity

v: v = c √μ = c t 2L 2 (Eq. 2.1.23.2)

where

c is the speed of light,

μ is the relative magnetic permeability of the soil (~1),

t is the travel time of the Time Domain Reflectometry (TDR) pulse,

2**L** is the travel path of the wave.

Since the travel time of electromagnetic waves through soil is a function of the effective moisture, and the relative contribution of water is a factor 20 times larger than all other (in)organic soil parts, the travel time is mainly a function of the water content.

Other alternative techniques such as satellite measurements became the practice in the event of regional or global investigations (Little et al., 1998).

Reagents

- None

Materials and equipment

For the gravimetric method:

- Oven with 105°C temperature
- Precision balance (±0.01 g)
- Aluminium tins
- Auger for soil sampling

TDR-based equipment for the in-situ determination of actual field capacity

- Portable or fixed equipment/data logger with TDR sensor(s)

Procedure

For the gravimetric method:

a. Weigh an aluminum tin, and record its weight (tare).
b. Place a soil sample of about 10g in the tin and record this weight as (wet soil + tare).
c. Place the sample in the oven at 105°C, and dry for 24 hours.
d. Weigh the sample, and record this weight as (dry soil + tare).
e. Return the sample to the oven and dry for several hours, and determine the weight of (dry soil + tare).
f. Repeat step 5 until there is no difference between any two consecutive measurements of the weight of the dry soil and the tare (de Angelis, 2007).

For the in-situ TDR-based method:

a. Calibrate the TDR sensors for the soil types in the laboratory
b. Create soil sample series which have different moisture contents (e.g. 5, 10, 30, 40 %) gravimetrically determined
c. Push the sensor into undisturbed samples
d. Plot soil moisture (%) against the measured values (%) and determine its accurancy
e. (In the field) push the TDR-sensor into the surface of the soil horizon

Calculations

The soil moisture content (SWC) in dry weight basis may be calculated using the following formula:

$$SWC=((W_2-W_3)\ (W_3-W_1)^{-1})\ 100\ \ [\%] \quad \text{(Eq. 2.1.23.3)}$$

where

W$_1$ is the weight of the tin [M; g]

W$_2$ is the weight of the moist soil + tin [M; g]

W$_3$ is the weight of the dry soil + tin [M; g]

The TDR equipment shows the results as percentages.

Remarks

- Methodological problems: the site is partially destroyed by the gravimetric method; the method itself modifies soil moisture distribution in time and space.
- Sample size is influenced by mean moisture content, the level of saturation, and the amount of insolation.
- Water content is not uniform throughout the profile.
- In the case of the in-situ TDR method, the soil water content will be measured using the different sensors installed at differing depths in the soil. A borer will be used in the placing of the sensors, seeking to make close contact between the sensor and the soil. Once the sensors have been installed they are connected to the Datalogger, with the corresponding program.

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24. Abundance and size of roots

Raúl Zornoza, Jose A. Acosta, Silvia Martínez, Virginia Sánchez-Navarro, Ángel Faz

Sustainable Use, Management, and Reclamation of Soil and Water Research Group, Department of Agrarian Science and Technology, Universidad Politécnica de Cartagena,

Paseo Alfonso XIII, 48, 30203, Cartagena, Spain

Importance and applications

Soil mechanical constraints can restrict the development of plant roots. High soil compaction by increasing machinery intensity can increase soil penetration resistance and bulk density, and as a consequence, decrease crop yields (Botta et al., 2004). The diagnosis of soil physical constraints by root development is mainly based on soil relative compaction, soil pore volume and soil penetration resistance (Micucci & Taboada, 2006). Thus, the assessment of the size and abundance of roots in the soils can help elucidate soil physical constraints for plant growth and development. The abundance of roots is also indicative of soil biological activity, and thus of a healthy soil.

Principle

The method proposed here is adapted from FAO, (2006). The size and abundance of plant roots is determined using a 10 cm x 10 cm transparent grid subdivided into 1 cm x 1 cm, as shown in Fig. 2.1.25.1. Soil is spread on a flat surface, preferably of a light colour, and the grid is placed over it to assess the abundance of roots. After this, roots are separated from the soil and the diameter measured with a ruler with the help of a magnifier, or with a magnifier with graticule for small roots.

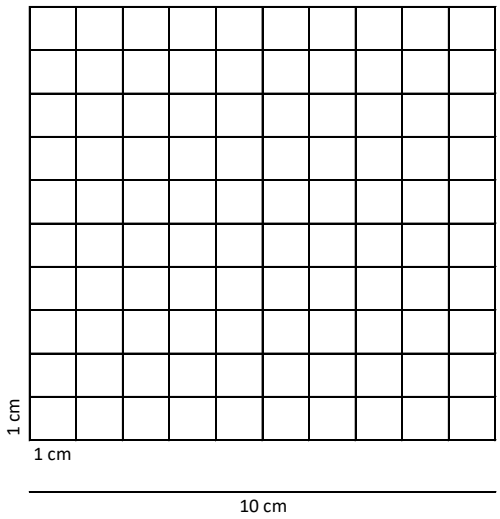

Figure 2.1.25.1. Grid for estimating abundance and size of roots

Reagents

- None

Materials and equipment

- 10 cm x 10 cm transparent grid subdivided into 1 cm x 1 cm. Material can be glass or plastic.
- Magnifier
- Magnifier with graticule for small roots
- Ruler for coarse roots

Procedure

a. Collect a soil sample from the layer or horizon to be characterised
b. Place the soil on a flat surface (preferably of a light colour). Spread the soil to cover all the surface
c. Put the 10 cm x 10 cm transparent grid on the soil
d. Record the number of roots present within the 10 cm x 10 cm grid. Record the number of roots according to two different categories for the classification of abundance: < 2 mm of diameter and > 2 mm diameter. A magnifier can be used to identify fine roots
e. Once the number of roots observed in the grid is recorded, separate the different roots from the soil and measure and record the size of the roots. For coarse roots, a ruler can be used with the help of a magnifier. For small roots, use a magnifier with graticule

Calculations

a. Abundance of roots: Indicate the number of roots < 2 mm diameter and the number of roots > 2 mm diameter and express this per 100 cm^2 (number or roots / 100 cm^2). Classify the abundance of roots according to Table 2.1.25.1:

Table 2.1.25.1. Classification of the abundance of roots (FAO, 2006)

Classification	Size of roots	
	< 2 mm diameter	> 2 mm diameter
None	0	0
Very few	1–20	1–2
Few	20–50	2–5
Common	50–200	5–20
Many	> 200	> 20

b. Classify the roots into four different size categories according to Table 2.1.25.2 (very fine, fine, medium, coarse). Calculate the percentage of roots belonging to each size category with relation to the total number of roots identified and measured.

Table 2.1.25.2 Classification of the size of roots (FAO, 2006)

Classification	Diameter (mm)
Very fine	< 0.5
Fine	0.5 – 2
Medium	2 - 5
Coarse	> 5

Remarks

- It is advisable to repeat the procedure at least three times per soil layer/horizon to obtain representative conditions regarding the layer/horizon.

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25. NH_4-Nitrogen

Alessandra Trinchera and Valentina Baratella

CREA – Council for Agricultural Research and Economics,

Research Centre for Agriculture and Environment, via della Navicella 2/4, 00184 Rome, Italy

Importance and applications

In the soil, the exchangeable ammonia (NH_4^+) is adsorbed on the exchange complex, while the nitrate form circulates in the liquid phase of the soil. Both the soil nitrate and the ammonia are mineral N forms contributing to crop nutrition, simultaneously present in a dynamic equilibrium: merely by virtue of this balance, they are under constant transformation from one form to another, as mediated by soil microflora (*Nitrobacter* and *Nitrosomonas* bacteria). The pool of exchangeable NH_4^+ is easily extractable from the soil by $CaCl_2$ or KCl extraction. It differs from the fixed NH_4^+, which is the pool of immobilised ammonia by the clay minerals, detectable only after soil treatment with fluoride acid.

The determination of ammonia (NH_4^+) is a key analysis on the assessment of soil fertility. Several methods are available for determination of this ion in soil extracts (Fig. 2.2.1.1). Among them, colorimetric methodologies by the Berthelot reaction (sometimes called the indophenol reaction) present the advantages of being quick, simple, and sensitive, and have been widely employed in the design of automated analyser systems (continuous flow analysis) (Keeney & Nelson, 1982; Mulvaney, 1996; Rhine et al., 1998). In agricultural sciences, the application of colorimetric methodologies is commonly found as a primary reaction methodology for the determination of NH_4^+ in plant materials (Davidson et al., 1970), soil and its extracts (Nelson, 1983), as well as fertilisers (Seely et al., 1967). It is used also in food, water, pharmaceuticals, and many others (Searle, 1984). As extraction solution, potassium chloride (KCl) is frequently adopted, because of its high determination coefficient.

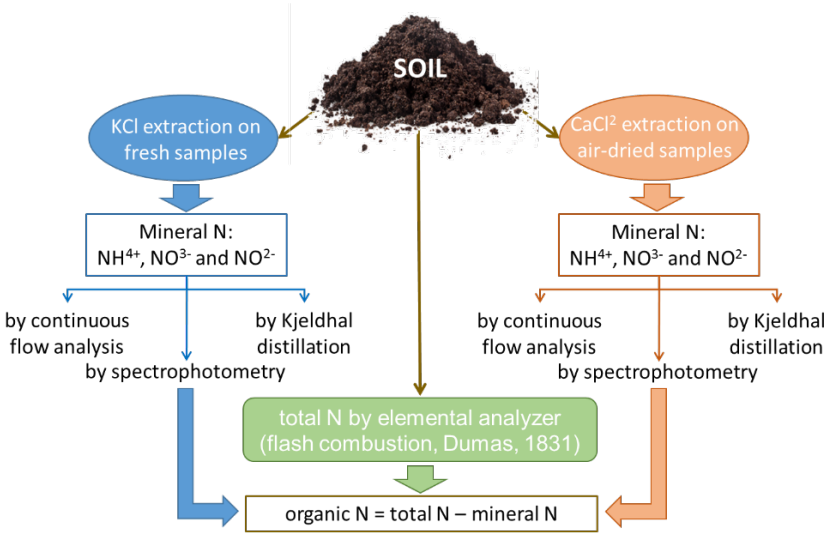

Figure 2.2.1.1 Diagram for the determination of the different soil nitrogen pools

Principle

The soil sample is treated with a solution of potassium chloride (2 mol x L^{-1}); the extract is then analysed by continuous flow colorimetric system. The ammonia nitrogen content is determined by the Berthelot reaction (sometimes called the “indophenol reaction”), discovered in 1859, in which sodium salicylate forms an indophenol in the presence of ammonia and hypochlorite. When NH_3, phenol, and hypochlorite were mixed in sequence, the reaction produced a blue or blue-green coloured solution, whose intensity is correlated to the concentration of NH_4^+ in the soil extract.

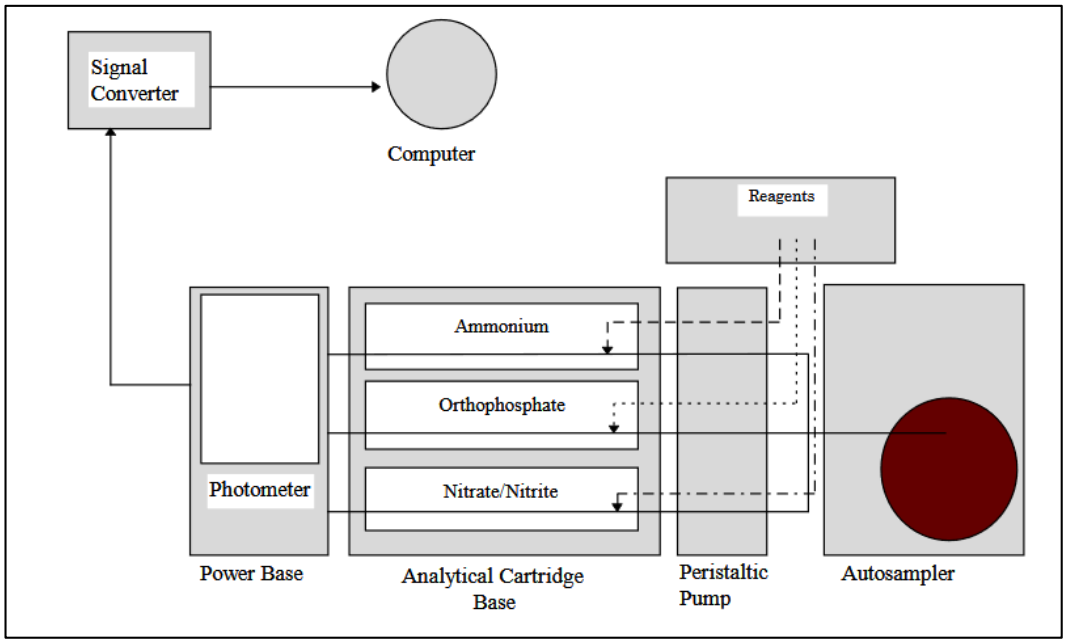

Figure 2.2.1.2 Operating scheme of the AutoAnalyzer Unit

Reagents

- Solution of potassium chloride (2 mol x L^{-1} KCl) (R0): dissolve 149 g of potassium chloride (KCl) in a 1000 mL graduated flask containing approximately 800 mL of H_2O. Bring to volume
- Buffer solution pH 5.2 (R1): dissolve 24 g of sodium citrate ($C_6H_5Na_3O_7$) and 33 g of sodium and potassium tartrate ($C_4H_4KNaO_6$) in H_2O in a 1000 mL graduated flask. Bring to volume
- Colorimetric reagent (R2): dissolve 80 g of sodium salicylate ($C_7H_5NaO_3$) and 25 g of sodium hydroxide (NaOH) in H_2O in a 1000 mL graduated flask. Bring to volume
- Sodium nitroprusside solution (R3): dissolve 1g of sodium nitroprusside dihydrate [Na_2Fe (CN) 5NO] x $2H_2O$ in a little water in a 1000 mL graduated flask. Bring to volume
- Sodium dichloroisocyanurate solution (R4): dissolve 2 g of sodium dichloroisocyanurate dihydrate ($C_3Cl_2N_3NaO_3$ x $2H_2O$ and 25 g of sodium hydroxide (NaOH) in H_2O in a 1000 mL graduated flask. Bring to volume
- Solution (200 mg x L^{-1}) of ammonia nitrogen (N-NH_4^+) (R5): dissolve 0.9439 g of ammonia sulphate [$(NH_4)2SO_4$] in H_2O in a 1000 mL graduated flask. Bring to volume. This solution can be stored for 1 month at a temperature of 4°C

- Standard working solutions of ammonia nitrogen (R6): prepare from R5 a series of standards containing from 0 to 1.6 mg x L^{-1} of $N\text{-}NH_4^+$ in 100 mL calibrated flasks. Bring to volume with potassium chloride (KCl) solution (2 mol x L^{-1}). Standard work solutions must be prepared at the time of use

Materials and equipment

- AutoAnalyzer Unit consisting of sampler, manifold or analytical cartridge, proportioning pump, heating cell, colorimeter equipped with tubular flow cell and 630-660 nm filters, recorder (Fig. 2.2.1.1)
- Plastic containers that do not absorb or release ammonia or nitrite ions
- Rotary agitator (40 rpm x $minute^{-1}$) or oscillating agitator (120-140 cycles x $minute^{-1}$)
- Common laboratory equipment

Procedure

o. Homogenise the soil sample, either manually or mechanically. The soil sample must be transferred to the laboratory in a refrigerator container. If the sample is analysed within three days from sampling, it can be stored at 4°C. Otherwise, to avoid possible loss in mineral nitrogen, it is necessary to freeze it at - 20°C. Temperature and duration of the defrosting process must be controlled: the samples should be maintained at room temperature, if they are analysed within 4 hours after removal from the freezer. It is also possible to maintain the samples at 4°C, in which case the defrosting time must not exceed 48 hours.

p. Extraction (Bremner & Keeney, 1966): transfer 20 g of soil sample to a 500 mL plastic container. Add 200 mL of R0 solution kept at a temperature of 20°C (the ratio must be 1:10). Keep stirring for 1 hour at 20°C. Centrifuge approximately 60 ml of the suspension for 10 min at approximately 3000 rpm min^{-1}. Transfer the supernatant into an Erlenmeyer flask. The content of nitrate, nitrite and ammonia ions should be determined within 24 hours, if not, store the extracts for no more than one week at a temperature not exceeding 4°C. Prepare the blank by following the same operating procedures, omitting the soil sample.

q. Moisture determination: weigh 20 g of the soil sample, set it into a preheated oven at 105°C for at least 16 hours. After cooling in the desiccator, weigh and calculate the moisture content in g x kg^{-1}.

r. Before starting the analysis, solutions (R1-R2-R3-R4) must be injected into the tubular flow cell until the absorbance value at λ = 660 nm becomes constant.

s. Make the calibration curve using the standard working solutions R6 (from 0 to 1.6 mg x L^{-1} of $N\text{-}NH_4^+$).

t. Perform the colorimetric analysis, according to the scheme shown in Figure 1, of the soil extracts in the solution of potassium chloride R0. Check the calibration every 10-20 samples, using the standard working solutions R6. If necessary, make a new calibration curve.

Calculations

The result is generally expressed as the ammonia nitrogen content ($N\text{-}NH_4^+$), expressed in mg x kg^{-1}:

$$C = \frac{(A - B) * D * V}{m} \qquad \text{(Eq. 2.2.1.1)}$$

where

C is the soil ammonia nitrogen content ($N\text{-}NH_4^+$), expressed in mg x kg^{-1};

A is the ammonia nitrogen content of the soil extracts, expressed in mg x L^{-1};
B is the ammonia nitrogen content of the blank sample, expressed in mg x L^{-1};
D is the dilution factor (D = 1, if no dilutions are made)
V is the extracts volume, expressed in millilitres mL
m is the soil mass, expressed in grams g

Remarks

- As reported, the soil ammonia is present in a dynamic equilibrium with nitrate. Thus, it is not useful to refer to a given range of soil N-NH_4^+, since it is affected by pedo-climatic and environmental conditions (temperature and soil moisture), fertilisation mode (mineral, organic), crop phenological phase, etc.
- The extraction temperature is a parameter that must be strictly reported in the analysis report (the amount of extractable ammonia nitrogen is influenced by the temperature). Centrifugation is preferred to filtration given that most paper filters can contain or absorb ammonia ions.
- The reaction is pH-dependent, therefore it is advisable to keep the alkaline reagents in plastic containers, with hermetic seals, to avoid the absorption of atmospheric CO_2.
- The presence of amino acids and proteins in solution can inhibit the reaction because these molecules react with sodium dichloroisocyanurate, by consequently decreasing the concentration of the hypochlorite in solution. Other N organic compounds can also react directly with hypochlorite.
- Copper (Cu) and mercury (Hg) can cause reaction inhibition, but the buffer solution limits their interference. Sulphur (S), selenium (Se) and halogens (X^-), in particular bromine (Br^-), can also interfere.

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26. NO_3- and NO_2-Nitrogen

Alessandra Trinchera and Valentina Baratella

CREA – Council for Agricultural Research and Economics,

Research Centre for Agriculture and Environment, via della Navicella 2/4, 00184 Rome, Italy

Importance and applications

Soil nitrate (NO_3^-) is an indicator of chemical soil fertility, being promptly utilised by plants (Keeney & Nelson, 1982). Due to potential N leaching in some specific pedo-climatic conditions, the use of high mineral N inputs or non-stabilised animal manure could determine an excess of NO_3^- in the soil circulating solution and, thus, consequent water pollution. Moreover, soil nitrite (NO_2^-) is seldom present: its determination is normally unwarranted except in neutral to alkaline soils receiving NH_4^- or NH_4^--producing fertilisers. When accumulated, or transformed into NO and NO_2, by interaction with other soil constituents, it could cause tropospheric ozone formation, acid rain, the greenhouse effect and the destruction of the stratospheric ozone (Van Cleemput & Samater, 1996; Su et al., 2011).

Several methods are available for the determination of nitrate (NO_3^-) and nitrite (NO_2^-) in soil extracts, such as key analysis on the assessment of soil fertility. Among them, colorimetric methodologies are simple and sensitive, being widely employed in the design of automated analyser systems (continuous flow analysis) (Keeney & Nelson, 1982; Mulvaney, 1996; Rhine et al., 1998).

Principle

The use of colorimetric methodologies for quantification of NO_2^- as NO_3^- and N-NO_2^- + NO_3^- by the reaction of Griess-Ilosvay (Dorich & Nelson, 1984; Keeney & Nelson, 1982; Nelson, 1983) showed the best performance by reducing preliminary the NO_2^- to NO_3^- (Mulvaney, 1996; Shinn 1941). It is recommended to use potassium chloride (KCl) as the extraction solution, which is already widely used in analytical laboratories because of its high determination coefficient (Fig. 2.2.2.1).

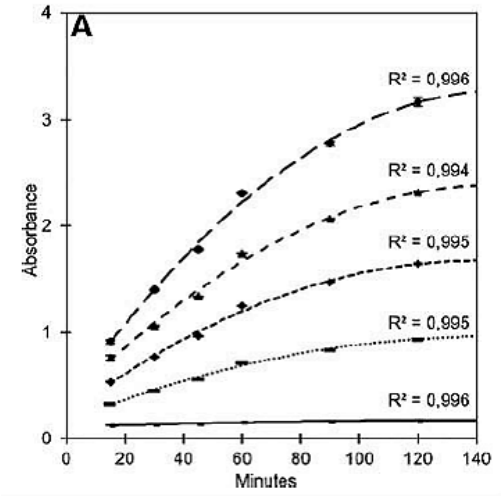

Figure 2.2.2.1 Tendency of N-NO_3^- readings for standard samples (quadratic model) in concentrations of 0.0, 2.5, 5.0, 7.5, and 10.0 mg L^{-1} for different reaction times (15, 30, 45, 60, 90, and 120 min) in 2 mol L^{-1} KCl. Symbols represent mean values, while vertical bars represent the standard error of the mean (n = 18) (after Sattolo et al., 2016)

The soil is treated with a solution of potassium chloride (2 mol x L^{-1}), then the extract is analysed by continuous flow colorimetry: the nitrate and nitrite content is determined by the Griess-Ilosvay reaction, in which nitrous and nitric ions form, by di-azotation with sulfonyl amide and N-(1-naphthyl)-ethylenediamine dihydrochloride, a reddish-purple compound whose intensity is measured at λ = 540 nm.

- Solution of N-(1-naphthyl)-ethylenediamine dihydrochloride (R6): dissolve 1 g of N-(1-naphthyl)-ethylenediamine dihydrochloride ($C_{12}H_{14}N_2$ x 2HCl) in H_2O, in a volumetric flask of 1000 mL, bring to volume with H_2O. This solution can be stored in the refrigerator, in a dark glass bottle, for no more than one week.
- Solution (100 µg x mL^{-1}) of nitric nitrogen ($N\text{-}NO_3^-$) (R7): dissolve 0.7218 g of potassium nitrate (KNO_3) in H_2O, in a volumetric flask of 1000 mL, bring to volume with H_2O. Keep the solution in a refrigerator.
- Standard working solutions of nitric nitrogen (R8): take 0, 1, 10, 50 and 100 mL of the solution R7 and transfer to five volumetric flasks of 1000 mL, bring to volume with the solution R0. In each of the four solutions, the nitric nitrogen concentration ($N\text{-}NO_3^-$) is, respectively, 0, 0.1, 1, 5 and 10 µg x mL^{-1}. Standard solutions must be prepared for each series of determinations.

Materials and equipment

a. AutoAnalyzer Unit consisting of sampler, manifold or analytical cartridge, proportioning pump, heating cell, colorimeter equipped with tubular flow cell and 630-660 nm filters, recorder (Fig. 2.2.2.2)

b. Plastic containers that do not absorb or release ammonium or nitrite ions

c. Rotary agitator (40 rpm x min^{-1}) or oscillating agitator (120-140 cycles x min^{-1})

d. Common laboratory equipment

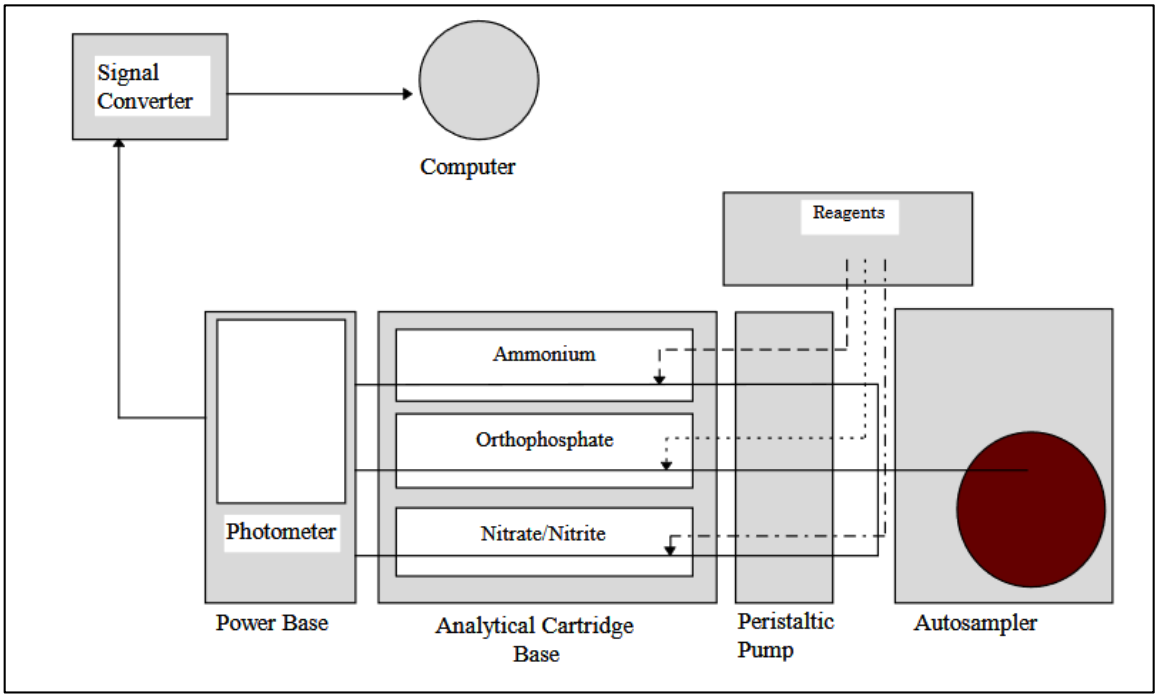

Figure 2.2.2.2 Operating scheme of the AutoAnalyzer Unit

Reagents

- Solution of potassium chloride (2 mol x L^{-1} KCl) (R1): dissolve 149 g of potassium chloride (KCl) in a 1000 mL graduated flask containing approximately 800 mL of H_2O. Bring to volume
- Solution (1: 1 v / v) of hydrochloric acid: add 500 mL of hydrochloric acid (HCl) 37% (R2) to a 1000 mL graduated flask containing approximately 450 mL of H_2O, mix and, after cooling, bring to volume with H_2O
- Ammonium hydroxide solution (100 mL x L-1) (R3): transfer 100 mL of ammonium hydroxide solution (NH_4OH) 30% to a 1000 mL graduated flask containing about 600 mL of H_2O, bring to volume with H_2O
- Buffer solution (R4): dissolve 53.5 g of ammonium chloride (NH_4Cl) in a 1000 mL graduated flask containing about 600 mL of H_2O, bring the pH of the solution to 8.5 by progressively adding R4, bring to volume with H_2O
- Solution of sulfonyl amide (R5): dissolve 10 g of sulfonyl amide ($C_6H_8N_2O_2S$) in a 1000 mL graduated flask containing approximately 300 mL of H_2O and 26 mL of hydrochloric acid (HCl) 37%, bring to volume with H_2O. Keep the solution in a refrigerator

Procedure

a. Homogenise the soil sample, either manually or mechanically. The soil sample must be transferred to the laboratory in a refrigerator container. If the sample is analysed within 3 days from sampling, it can be stored at 4°C. Otherwise, to avoid possible losses in mineral nitrogen, it is necessary to freeze it at -20°C. The temperature and duration of the defrosting process must be controlled: the samples should be thawed at room temperature if they are analysed within 4 hours after removal from the freezer. It is also possible to thaw the samples at 4°C, in which case the defrosting time must not exceed 48 hours

b. Extraction (Bremner & Keeney, 1966): transfer 20 g of soil sample to a 500 mL plastic container. Add 200 mL of R1 solution kept at a temperature of 20°C (the ratio must be 1:10). Keep stirring for 1 hour at 20°C. Centrifuge approximately 60 ml of the suspension for 10 min at approximately 3000 rpm min^{-1}. Transfer the supernatant to an Erlenmeyer flask. The content of nitrate, nitrite and ammonium ions should be determined within 24 hours, if not, store the extracts for no more than one week at a temperature not exceeding 4°C. Prepare the blank by following the same operating procedures, omitting the soil sample

c. Moisture determination: weigh 20 g of the soil sample, set it into a preheated oven at 105°C for at least 16 hours. After cooling in the desiccator, weigh and calculate the moisture content in g x kg^{-1}

d. Before starting the analysis, solutions (R4-R5-R6) must be injected into the tubular flow cell until the absorbance value at λ = 540 nm becomes constant

e. Make the calibration curve using the standard working solutions R8 (from 0 to 1.6 mg x L^{-1} of $N\text{-}NH_4^+$)

f. Perform the colorimetric analysis, according to the scheme shown in Figure 1, of the soil extracts in the solution of potassium chloride R1. Check the calibration every 10-20 samples, using the standard working solutions R8. If necessary, make a new calibration curve

Calculations

The result is generally expressed as the nitrate and nitrite nitrogen content ($N\text{-}NO_2^-$ + $N\text{-}NO_3^-$), expressed in mg x kg^{-1}:

$$C = \frac{(A - B) * D * V}{m} \qquad \text{(Eq. 2.2.2.1)}$$

where

C is the soil nitrate and nitrite nitrogen content ($N\text{-}NO_2^-$ + $N\text{-}NO_3^-$) [mg kg^{-1}],
A is the nitrate and nitrite nitrogen content of the soil extracts [mg L^{-1}],
B is the nitrate and nitrite nitrogen content of the blank sample [mg L^{-1}],
D is the dilution factor (D = 1 if no dilutions are made)
V is the extracts volume [mL]
m is the soil mass [g]

Remarks

- The soil mineral N content, as nitrate, nitrite and ammonia, represents the pool of nitrogen available to the crop. It is useful to refer to a given range of soil $N\text{-}NO_3^-$ and $N\text{-}NO_2^-$ related to land use or soil type, with these values being dynamic and strongly affected by pedo-climatic and environmental conditions (temperature and soil moisture), fertilisation mode (mineral, organic), crop phenological phase, etc.
- Interference may be due to the presence of coloured components in the sample, which can absorb at the wavelength used. Other interferences may be related to the presence in the sample of strong oxidants or reducing agents, at high concentrations of aromatic amines, copper (Cu), iodine (I) and humic acids.
- The Griess-Ilosvay method is very sensitive and specific and is not affected by cations and anions interference. The soil extracts in R1 may on occasions be coloured, but this occurrence does not interfere with the analysis, according to the method.
- Colour development is very rapid. At 25°C, the maximum colouring is achieved in 10 minutes and remains stable for a few hours.

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27. Available P content by the Olsen method

Alessandra Trinchera and Valentina Baratella

CREA – Council for Agricultural Research and Economics,

Research Centre for Agriculture and Environment, via della Navicella 2/4, 00184 Rome, Italy

Importance and applications

As essential element in the life cycle of plants, P constitutes 2 to 3% of the tillable soil profile, where it is strongly bound to calcium, aluminium, iron and other elements as a phosphate anion (PO_4^{3-}). Phosphorus is present in the soil in relatively low quantities, between 0.2 and 5 g Kg^{-1}. From the point of view of plant nutrition, the soil phosphates can be divided into three fractions in equilibrium:

1) phosphates present in the liquid phase;

2) phosphates in labile form;

3) phosphates in non-labile form (Fig. 2.2.3.1).

In the soil solution, P ranges from 0.01 to 0.2 mg / L, and is not very mobile. Two different methods for the analysis of soil available P content will be illustrated in the following paragraphs: the Olsen method (Section 2.2.3.1), and the Mehlich 3 method (Section 2.2.3.2).

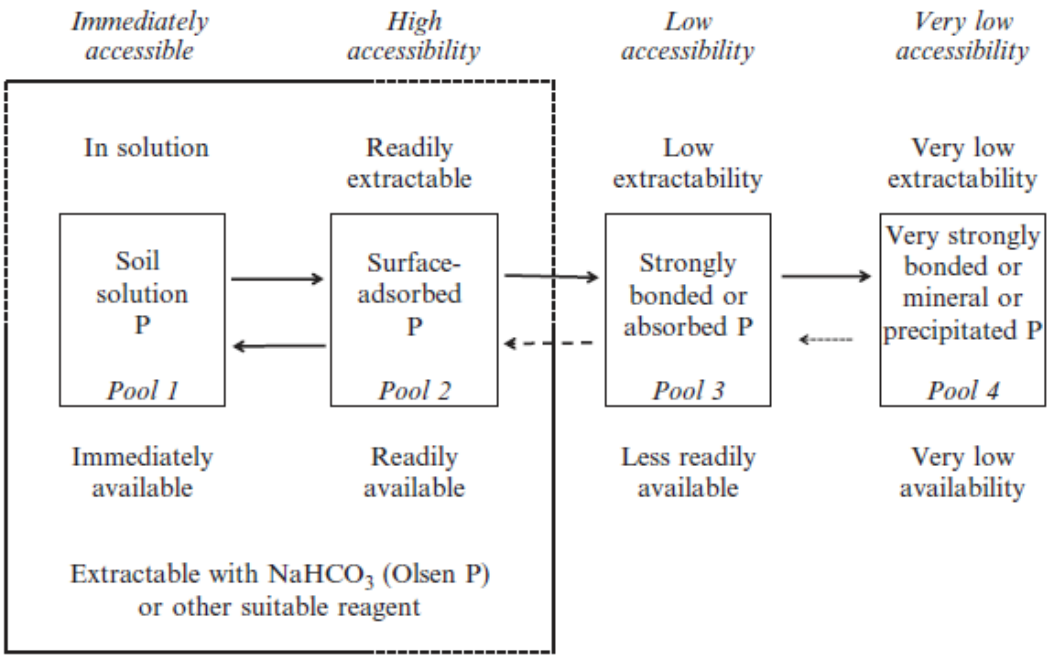

Figure 2.2.3.1 Forms of inorganic P in soils in terms of accessibility, extractability, and plant availability, in relation to the extraction range of the Olsen method (from Johnston et al., 2014)

The analysis of assimilable P content is critically important to the discussion about the retention of plant-available P in soil. To evaluate the P available for the crops in calcareous / neutral soils, Kamprath and Watson (1980) proposed the extraction with diluted solutions of weak acids and buffered alkaline solutions (Olsen et al., 1954; Soltanpour & Schwab, 1977). The Olsen method is safely suitable for a wide range of soil types and pH values. In acid soils containing Al and Fe phosphate, the P concentration in the solution increases as the pH rises. Precipitation reactions in acid and calcareous soils are reduced to a minimum because the concentrations of Al, Ca and Fe remain at a low level in this extractant.

In long-term experiments (> 40 years at Rothamsted, Woburn and Saxmundham), a linear relationship was demonstrated between the increase in Olsen P and the increase in total soil P (when both are expressed in kg P ha^{-1}) (Fig. 2.2.3.2) (Johnston et al., 2014).

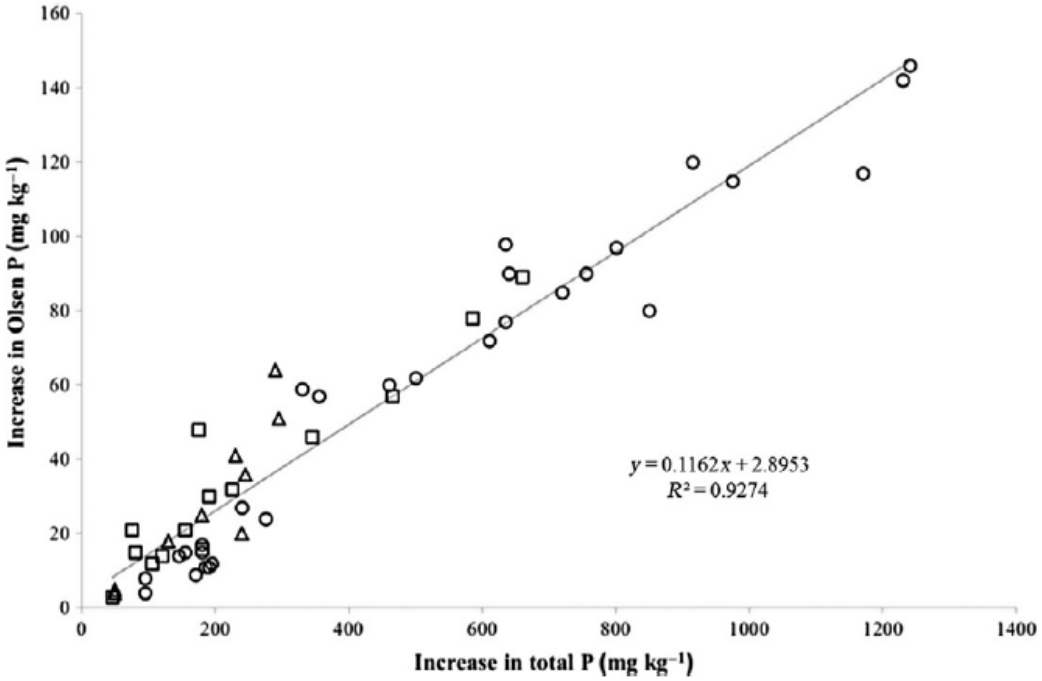

Figure 2.2.3.2 Relationship between the Olsen P and the total P in soils of long-term experiments (>40 years) where P has been applied as both fertiliser and organic manure: silty clay loam ○), sandy loam (□), and sandy clay loam (△) (from Johnston et al., 2014).

A relationship between crop yield and Olsen P can be profitably described by an asymptotic regression equation (Mitscherlich type) and the asymptotic (maximum) yield determined. From this relationship, the critical Olsen P associated with the yield at an arbitrary proportion of the asymptotic yield can be calculated from the parameters of the fitted curve using an appropriate equation.

Principle

According to the method, the soil is extracted with 0.5 mol solution of sodium bicarbonate at pH 8.5. In calcareous, alkaline or neutral soils containing calcium phosphate, this extracting solution decreases the concentration of Ca in solution by precipitating Ca as $CaCO_3$, and the result is an increase of the P concentration in the solution. The concentration of phosphorus in the solutions obtained is then generally determined by the colorimetric method.

Reagents

- Solution of sulphuric acid (2.5 mol x L^{-1}) (R1): carefully add 140 ml of sulphuric acid (H_2SO_4) [96%] to a 1000 mL graduated flask containing approximately 5.00 mL of H_2O. Stir and, after cooling, dilute to volume with H_2O.
- Sodium hydroxide solution (1.0 mol x L^{-1}) (R2): dissolve 40 g of sodium hydroxide (NaOH) in H_2O in a 1000 mL graduated flask. Stir and, after cooling, dilute to volume with H_2O.
- Sodium bicarbonate solution (0.5 mol x L^{-1}) (R3): dissolve 42 g of sodium bicarbonate ($NaHCO_3$) in a beaker containing about 900 mL of H_2O. Bring the pH to 8.5 by adding the R2 solution drop by drop. Transfer to a 1000 mL graduated flask and dilute to volume with H_2O. To avoid direct contact of the solution with atmospheric air, add a layer of mineral oil.
- Activated carbon: check the purity of this reagent by performing an extraction with R3. In the presence of phosphorus, wash several times with R3 up to levels of P that are not detectable by spectrophotometry.
- P-nitrophenol solution (0.25%) (R4): in a 100 mL volumetric flask, dissolve 0.25 g of p-nitrophenol ($NO_2C_6H_4OH$) in H_2O.
- Ammonium molybdate solution (40 g x L^{-1}) (R5): dissolve 40 g of ammonium molybdate [$(NH_4)\cdot 6Mo_7O_{24}$ x $4H_2O$] in H_2O in a 1000 mL graduated flask. Dilute to volume with H_2O and store in a dark glass container.
- Potassium tartrate antimony solution (1 mg of Sb x mL^{-1}) (R6): dissolve 0.2728 g of potassium antimony tartrate [(K (SbO) x $C_4H_4O_6$ x ½ H_2O] in H_2O in a 100 mL graduated flask. Bring to volume with H_2O.
- Solution of ascorbic acid (0.1 moles x L^{-1}) (R7): dissolve 1.76 g of ascorbic acid ($C_6H_4O_6$) in H_2O in a 100 mL graduated flask. Bring to volume with H_2O. Prepare the solution at the time of use.
- Sulphomolybdic Reagent (R8): mix, at the time of use, 50 mL of the R1 solution, 15 mL of the R5 solution, 30 mL of the R7 solution and 5 mL of the R6 solution.
- Standard P solution (1000 mg x V) (R9): transfer 4,3938 g of potassium dihydrogen phosphate (KH_2PO_4) dried in an oven at 40°C to a 1000 mL graduated flask containing approximately 500 mL of H_2O. After dissolving the salt, dilute to volume with H_2O.
- Diluted standard P solution (R10): transfer 10 mL of R9 into a 1000 mL graduated flask. Bring to volume with H_2O. In this solution the phosphorus concentration is 10 mg x L^{-1}.

Materials and equipment

- pH meter with temperature compensator
- Oscillating agitator at 120-140 cycles x $minute^{-1}$
- 0.45 µm membrane filters
- Spectrophotometer
- Common laboratory equipment

Procedure

a. *Extraction*: transfer 2 g of the sample to a conical flask or a 125 mL plastic container. Add 0.5 g of activated carbon and 40 mL (V1) of the R3 solution. Stir for 30 minutes and filter several times with Whatman n° 42. If necessary, use the 0.45 µm membrane filter. Prepare the blank test by following the same operating procedures, but omitting the soil sample

b. *Colorimetric determination*: transfer an aliquot of the clear, extracted solution containing from 2 to 40 µg of P (V2) to a 50 mL graduated flask. Add 5 drops of the R4 solution and, drop by drop, a quantity of the R1 solution to turn the colour of the indicator to yellow. Dilute with H_2O to approximately 25 mL and add

8 mL of the R8 reagent. Bring to volume with H_2O. After 10 minutes, read the extinction value 882 nm on the spectrophotometer against a blank containing all the reagents excluding the phosphorus solution

c. *Calibration curve*: transfer 0, 5, 10, 15, 20 and 25 mL of the R10 standard solution to six graduated flasks (50 mL volume). Dilute with H_2O to approximately 25 mL and add 8 mL of the R8 reagent. Bring to volume with H_2O. In each of the six solutions, the phosphorus concentration is, respectively: 0, 1, 2, 3, 4 and 5 mg x L^{-1}. After 10 minutes, read the extinction value 882 nm on the spectrophotometer against a blank containing all the reagents excluding the phosphorus solution.

Calculations

The result is generally expressed as phosphorus content, expressed in mg x kg^{-1}:

$$C = (\mathrm{A} - \mathrm{B}) \frac{V_1}{V_2} \frac{50}{m} \qquad \text{(Eq. 2.2.3.1.2)}$$

where

C is the soil extractable P content [mg kg^{-1}],

A is the P concentration in the sample solution [mg L^{-1}],

B is the P concentration in the blank sample solution [mg L^{-1}],

V_1 is the volume of the extract [40 mL],

V_2 is the volume of the sample solution used for colorimetric determination,

m is the soil mass [g]

Table 2.2.3.1 Range of values for P-Olsen data clustered according to major FAO soil groups (FAO, 1988; after Batjes, 2010)

P-Olsen (mg kg^{-1})	pH (in water)	TOC (g kg^{-1})	N_{tot} (g kg^{-1})	Soil type (0-20 cm)
7.2474	6.9183	2.7425	0.4114	Arenosols
19.528	8.0784	6.1143	1.2413	Calcisols
12.078	7.9485	7.5033	1.9959	Cambisols
13.061	7.9306	7.7798	1.3277	Fluvisols
10.638	7.0000	15.858	0.4100	Gleysols
13.381	7.0114	5.0595	1.6009	Luvisols
16.739	7.5408	4.3386	0.8795	Regosols
8.5943	7.2343	6.5474	0.9522	Vertisols

Remarks

- All the products used must be free of silicon, taking into account the reactivity of this element with the sulphomolybdic reagent. For the same reason, it is preferable to use distilled water since deionised water can contain silica.
- The presence of sodium bicarbonate, carbonate and hydroxyl ions in the solution lowers the activity of Ca^{2+} and Al^{3+} with a consequent increase in the solubility of phosphorus (P).
- In calcareous soils, the increased solubility of calcium phosphate derives from the decrease in the calcium concentration due to the high presence of carbonate ions and the consequent precipitation of $CaCO_3$.
- At high pH, the increase in negative charges and / or the decrease of the adsorption sites on the surfaces of aluminium and iron oxides can lead to the desorption of the fixed phosphorus.
- In acid or neutral soils, the solubility of aluminium and iron phosphates is increased by the increase in the concentration of hydroxyl ions which induces a decrease in the concentration of Al^{3+} with the formation of aluminate ions, and of Fe^{3+}, with precipitation of oxides.

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28. Available P content by Mehlich 3 method and ICP-AES

Valentina Baratella and Alessandra Trinchera

CREA – Council for Agricultural Research and Economics,

Research Centre for Agriculture and Environment, via della Navicella 2/4, 00184 Rome, Italy

Importance and applications

The Mehlich 3 index of phosphorus availability (M3-P) (Mehlich, 1984) measures the readily plant-available P of the soil solution. The M3 method is widely used in North America, Europe, and Australia since it can be applied to determine the nutrient status of soils ranging in reaction from acid to basic, and particularly for assessing available P and K (Jones, 1998; Zbiral & Nemec, 2000a,b; Cox, 2001; Bolland et al., 2003). Many studies in the literature provide a strong correlation between M3-P and crop ecophysiological responses, i.e. plant uptake and plant yield, for a wide range of soils (Tran & Giroux, 1987; Ziadi et al., 2001; Mallarino, 2003). The method has been the only soil test validated through inter-laboratory studies for the extraction of plant-available P, which is usually less than 0.01 – 0.02% of the total P, and has been used as a reference method for testing soils for extractable P (Alvey, 2013 and references therein; Zhang et al., 2009). A comparison of the M3 method with the many other methods developed to determine the soil P content is given in Fig. 2.2.3.3.

Name of Test	Extractant	Reference
AB-DPTA	1M NH_4HCO_3 + 0.005 M DPTA, pH 5	59
Bray I	0.025 N HCl + 0.03 N NH_4F	6
Bray II	0.1 N HCL + 0.03 N NH_4F	6
Citric acid	1% Citric acid	3
EDTA	0.02 M Na_2-EDTA	61
Mehlich 1	0.05 M HCl + 0.0125 M H_2SO_4	224
Mehlich 3	0.015 M NH_4F + 0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3	56
Morgan[a]	0.54 N HOAc + 0.7 N NaOAc, pH4	5
Olsen	0.5 M $NaHCO_3$, pH 8.5	58
Truog	0.001 M H_2SO_4 + $(NH4)_2SO_4$, pH 3	4
Water[b]	Water	225

[a]A modification of the Morgan by Wolf to include 0.18 g/L DPTA gives better correlations for micronutrients.
[b]From: C.A. Sanchez. Soil Testing and Fertilizer Recommendations for Crop Production on Organic Soils in Florida. University of Florida Agricultural Experiment Station Bulletin 876, Gainesville, 1990.

Chemical	Form of Phosphorus Extracted
Acid (H^+)	Solubilizes all chemical P in the following order Ca-P>Al-P>Fe-P
Bases (OH^-)	Solubilizes Fe-P and Al-P in respective order. Also results in release of some organic P
Fluoride ion	Forms complexes with Al thus releasing Al-P. Also precipitates Ca as CaF_2 and thus will extract more Ca-P as $CaHPO_4$. No effect on basic Ca-P and Fe-P
Bicarbonate ions	Precipitate Ca as $CaCO_3$ thus increasing solubility of Ca-P. Also remove Al-bound P
Acetate ions	Form weak complexes with polyvalent metal ions. Possibly prevents readsorption of P removed by other ions
Sulfate ions	Appear to reduce readsorption of P replaced by H ions

[a]Adapted from G.W. Thomas and D.E. Peaslee, in *Soil Testing and Plant Analysis*. Madison, WI: Soil Sci. Soc. Am. Inc., 1973 and E.J. Kamprath and M.E. Watson, in *The Role of Phosphorus In Agriculture*. American Society of Agronomy Inc. 677 South Segoe Road, Madison WI 53711, 1980.

Figure 2.2.3.3 Some historical and commonly used soil tests and extracting solutions for determining available phosphorous, and forms of phosphorus extracted (modified from Barker et al., 2015)

M3 correlates to Bray P1 (Bray & Kurtz, 1945) on acid soils (R^2 = 0.966) and to Olsen (Olsen et al., 1954) on alkaline soils (R^2 = 0.918), and to P extracted by M2, strontium chloride–citric acid, and water (Fig. 2.2.3.4) (Mehlich, 1984; Simard et al., 1991; Mallarino, 1995; Sawyer et al., 1999; Zbiral & Nemec, 2002; Iatrou et al., 2014). The Olsen and M3 tests are generally well correlated across all soils, the relationship can be slightly affected by the inclusion of calcareous soils (the slope of lines and intercepts tends to be similar for the different pH classes) (Mallarino, 1995). However, none of the three methods (Bray, Olsen and M3) correlate well for calcareous soils (Mallarino, 1995).

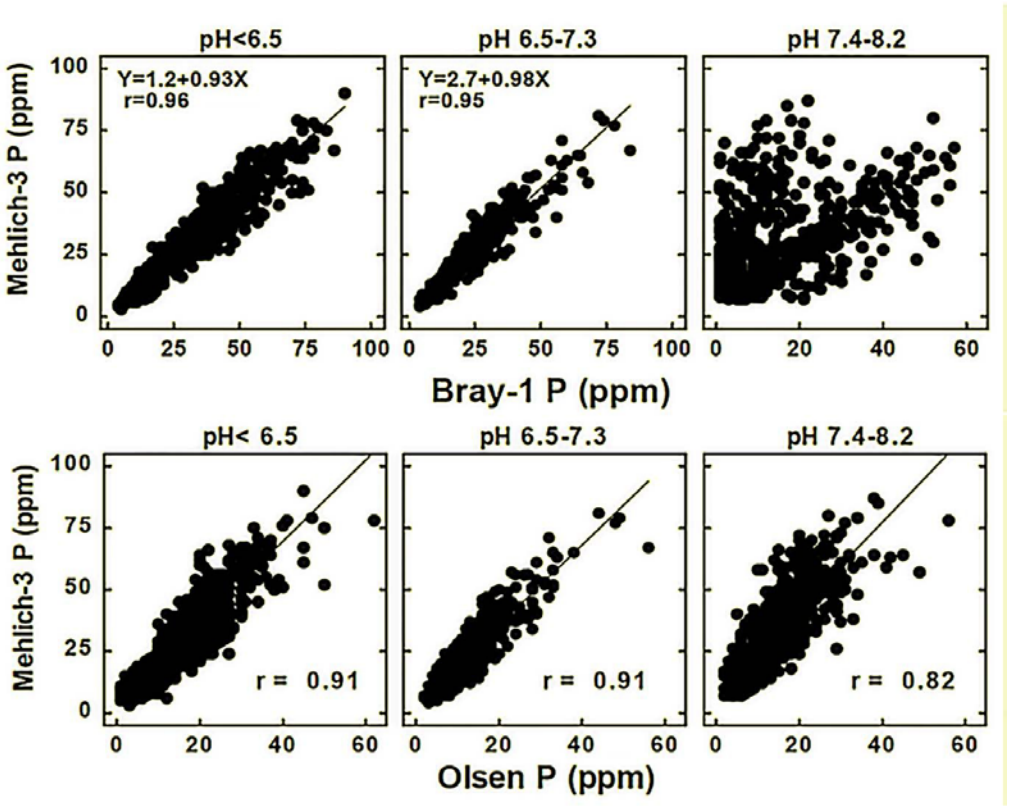

Figure 2.2.3.4 Correlations between amounts of P extracted by Mehlich3, Olsen and Bray tests for 2925 sample soils across acid, neutral, and high pH. Bray was strongly influenced by soil pH and extracted less P than the M3 in many calcareous soils (data points with Bray values near zero but higher M3 values). Olsen and M3 are well correlated across all soils and the correlation was highly independent of soil pH. The Olsen, as expected, extracts less P than the other tests (modified from Sawyer & Mallarino, 1999)

In addition to P, the extraction with M3 solution showed significant correlations with different currently used methods for K, Ca, Mg, Na, Cu, Zn, Mn, B, Al, and Fe (for a more detailed review, see Ziadi et al., 1993), it is therefore being widely used as the 'universal extractant' to evaluate the soil macro- and micro-nutrient status (Zhang et al., 2009, Schroder et al., 2010). Unlike the Olsen method, M3 extracts can be analysed by inductively coupled plasma emission spectroscopy (ICP), reducing the analysis time and also providing the advantage of measuring the P content simultaneously with the other nutrients in the same soil extract (Sawyer & Mallarino, 1999; Iatrou et al., 2014). Compared with the original colorimetric determination of M3 extracts (ascorbic acid method, considered specific for the orthophosphate form of P), the ICP usually measures higher values of M3-P (Mallarino,
2003; Sikora et al., 2005; Iatrou et al., 2014), since the instrument reads all P forms in the sample (the orthophosphate P form and also other small amounts of inorganic and simple organic P forms). However, several studies showed highly significant relationships between M3 ICP and colorimetric M3 for both acidic and alkaline soils (Figure 2.2.3.5) (Mallarino, 2003; Sikora et al., 2005; Pittman et al., 2005; Iatrou et al., 2014 and references therein). The reading difference between the colorimetric determination and the ICP analysis does not apply for the other nutrients that can be measured in M3 soil extracts (Mallarino, 2013).

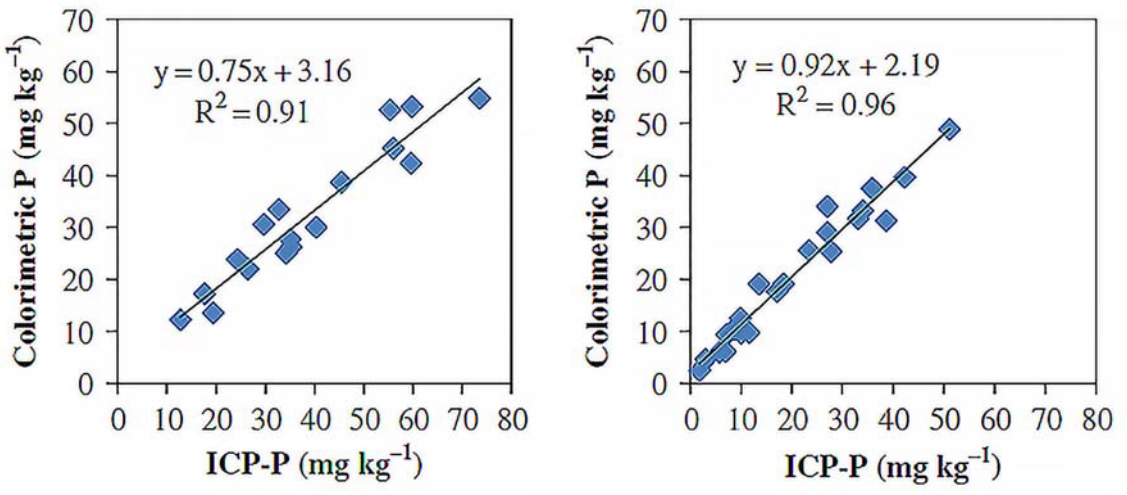

Figure 2.2.3.5 Relationships for extractable P content between M3 ICP test and colorimetric M3 test, for acidic soils (pH from 4.30 to 6.75, on the left) and for alkaline soils (pH from 7.12 to 7.98, on the right) (from Iatrou et al., 2014)

Principle

In the Mehlich 3 procedure, P extractable phosphorus is obtained by reaction with a dilute acid-fluoride-EDTA solution of pH 2.5. The extracting solution is composed of CH_3COOH (0.2 M), NH_4NO_3 (0.25 M), NH_4F (0.015 M), HNO_3 (0.013 M), and EDTA (ethylene diamine tetra-acetic acid 0.001 M). The phosphorus is solubilised under different mechanisms: nitric and acetic acids increase the solubility of Fe and Al phosphates and extracts Ca phosphates, fluoride increases the quantity of orthophosphate in solution by complexing Al cations, and the acetic acid keeps the solution buffered below pH 2.9 to prevent CaF_2 precipitate. The M3 extractant is less aggressive towards apatite and other calcium phosphates and is neutralised less by carbonate than the Bray extractant. The variety of M3 acids (i.e., acetic and nitric acids) makes it more versatile for soils having high concentrations of calcium. The M3 extracts are then analysed by inductively coupled plasma atomic emission spectrophotometer (ICP-AES) in radial mode, which allows multiple element determinations on the same soil extract. Data on the elemental concentration are reported as mg kg^{-1} soil.

The method is applicable for the determination of extractable K, Ca, Mg, Na and micronutrients, such as Mn, Fe, Cu and Zn. The exchangeable base cations K, Ca, Mg, and Na are removed by the action of ammonium nitrate and nitric acid, with a recovery nearly identical to the ammonium acetate method. The micronutrients are extracted by NH_4^+ and the chelating agent EDTA, their recovery is linearly related to DTPA and 0.1M HCl methods.

The repeatability and reproducibility of M3 for plant available macro- and micro-nutrients were thoroughly evaluated through inter-laboratory studies by Zhang et al., (2009) and Schroder et al., (2009).

Reagents

- Nitric acid 10% v/v: dilute 10 mL concentrated HNO_3 (HNO_3 68-70% ACS grade, CAS 7698-37-2) in 100 mL of deionised water
- Ammonium fluoride (NH_4F) CAS 12125-01-8
- Ethylene diamine tetra-acetic acid (EDTA) CAS 60-00-4
- Ammonium nitrate (NH_4NO_3) CAS 6484-52-2
- Glacial acetic acid (CH_3COOH) CAS 64-19-7

- M3 stock solution: (1.5 M NH_4F + 0.1 M EDTA): dissolve 55.56 g of ammonium fluoride (NH_4F) in 600 mL of deionised water in a 1 L volumetric flask. Add 29.23 g of EDTA to this mixture, dissolve, bring to 1 L volume using deionised water, mix thoroughly, and store in plastic bottle
- M3 extracting solution: dissolve 200.1 g of ammonium nitrate (NH_4NO_3) in a 10 L plastic carboy containing 8 L of deionised water, and add 100 mL of stock solution M3, 115 mL concentrated acetic acid (CH_3COOH), 82 mL of 10% v/v nitric acid, bring to 10 L with deionised water and mix thoroughly. The pH of the extracting solution should be 2.3+0.2. Store in a polyethylene container. Make a fresh solution weekly. Store in a refrigerator

Materials and equipment

- Oscillating shaker, 200 oscillations min^{-1}
- Centrifuge tubes, 50-mL, polyethylene or poly-propylene
- Centrifuge
- Filter paper, Whatman 42, 150 mm
- Pipettes, electronic digital, 1000 µL and 10 mL, with tips
- Inductively coupled plasma atomic emission spectrophotometer (ICP-AES)
- Common laboratory equipment

Procedure

a. Pre-rinse Whatman N42 filters: suspend filter funnels with filters on test-tube racks and fill filters with deionised water, let water drain completely from funnels and repeat using the M3 extracting solution.

b. Extraction: weigh 2.5 g of air-dry soil, 2 mm sieved, into a 50-mL centrifuge tube. Add 25.0 mL of the M3 extracting solution (soil:solution = 1:10). Shake immediately for 5 min at 200 oscillations min^{-1} at room temperature (20°C± 2°C). Centrifuge at 2000 rpm for 10 min or until the solution is free of soil mineral particles. Then decant, filter through pre-rinsed Whatman N42 filter paper until clear extracts are obtained, and collect the extracts into clean centrifuge tubes. A blank of M3 is prepared. Analyse by ICP-AES immediately after the extraction, or store at 4°C and analyse the samples within 72 h. Use the M3 extracting solution to dilute those samples with concentrations greater than the high standard.

c. Calibrations standards: these will vary depending on the expected soil P concentrations. From a 1,000 mg L^{-1} standard solution, prepare 1 L of the standard at the highest P concentrations using the M3 extracting solution for dilution. Then prepare 250 mL of the other calibration standards by diluting the most concentrated one. In general, make up 0, 10, 25, 50 mg Kg^{-1} calibration standards. Depending on the soil analysed, standards between 0 and 10 mg Kg^{-1} may be required for samples with low P concentrations.

d. Analysis: calibrate the ICP instrument using the calibration standards and following the manufacturer's recommendations, then analyse the samples. If a sample has P concentrations above the highest standard, dilution should be made using the M3 extracting solution.

Calculations

The result is generally expressed as soil M3 extractable P content, expressed in mg kg^{-1} and given directly by the ICP instrument.

Calculation of mg kg^{-1} of P in the soil is as follows:

$$P = \frac{C}{F}\frac{V}{W}DF \qquad \text{(Eq. 2.2.3.2)}$$

where

P is the soil extractable P content [mg kg^{-1}],

C is the sample P content from the ICP read-out [mg L^{-1} or µg L^{-1} for the ICAP Trace],

F is the concentration unit factor (e.g. 1.00 for ICAP61E, 1000 for ICAP Trace),

V is the final volume of the (undiluted) sample solution [mL],

W is the weight of the sample [g],

DF is dilution factor (DF = 1.00 with no sample dilution).

If dilution of the sample is required, the DF is given by

$$DF = \frac{B + C}{C} \qquad \text{(Eq. 2.2.3.3)}$$

where

B is mL of the acid blank matrix used for dilution,

C is mL of the sample aliquot taken for dilution,

B + **C** is the volume of (diluted) sample solution.

Soil-test P interpretation classes available in the literature for the Bray, Olsen, colorimetric M3, and M3-ICP tests are reported in Table 2.2.3.2 (Mallarino et al., 2013).

Table 2.2.3.2 Interpretation of M3-P soil test values measured by the Bray-P1, Olsen, colorimetric M3 and M3 ICP tests for most Iowa soils and crops (15-20 cm soil sampling depth) (modified from Mallarino et al., 2013).

Relative Level	P (g kg^{-1}) M3 ICP	P (g kg^{-1}) Bray P1 or M3 P	P (g kg^{-1}) Olsen
Very low	0–15	0–8	0–5
Low	16–25	9–15	6–9
Optimum	26–35	16–20	10–13
High	36–45	21–30	14–18
Very high	46+	31+	19+

Remarks

- Air-dried soils may be stored several months without affecting the M3-P measurement
- During extraction, since the shaking time is so short it is advisable to do the extraction in batches of samples (maybe 10 at a time). The idea is to have all samples in contact with the extracting solution the same amount of time.
- Mehlich (1984) proposed to use 0.2 % $AlCl_3$ as a rinsing solution for all labware, including qualitative filter paper. Ziadi et al., (1993) suggested the use of M3 extracting solution as a rinsing solution for filter paper.
- Because of Zn contamination, Pyrex glassware cannot be used for extraction or storage of the M3 extractant and laboratory standards. Tap water is a major source of Cu and Zn contamination.
- The M3 extract is not stable for long periods of time; the extracting solution should not be used after 10 days.
- The ICP analysis of M3 extracts has been reported to quantify higher P amount than colorimetric methods (Mallarino, 2003; Pittman et al., 2005; Iatrou et al., 2014), therefore caution is needed when comparing results.

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29. Potential and effective cation exchange capacity

Sören Thiele-Bruhn

Soil Science, University of Trier, Behringstr. 21, D-54286 Trier, Germany

Importance and applications

The cation exchange capacity (CEC) of soils is a chemical soil parameter of overall relevance. To a great extent, it determines the ability of soils to retain nutrients and toxic compounds as well as the ability to provide plants with nutrients. Thus, CEC is a key parameter of soil fertility as a major ecosystem service of soils. The CEC depends on the types and content of clay minerals and pedogenic oxides as well as soil organic matter and its quality. The contribution of functional groups to the CEC varies with pH so the potential CEC, measured at high pH >7, deviates more and more from the effective CEC (ECEC, measured at original soil pH) the more acidic a soil is. The potential CEC is furthermore needed to determine the soil base saturation. Numerous methods to determine the potential CEC and the ECEC can be found in the literature.

Two methods have been standardised (ISO 13536 and ISO 11260) to determine potential CEC and ECEC, and are reported here. These are ISO 1160 “Soil quality - Determination of effective cation exchange capacity and base saturation level using barium chloride solution” and ISO 13536 “Soil quality - Determination of the potential cation exchange capacity and exchangeable cations using barium chloride solution buffered at pH = 8.1” (ISO, 1995, 2018). The latter method is also needed to determine the base saturation.

Both guidelines were published several years ago, so not all details refer to the latest state of the art. Hence, some comments have been added to the text. These are suggestions for alternative realisation of the ISO guidelines.

Principle

The methods described here are largely based on the International Standard ISO 13536 (Section 2.2.4.1) and on the ISO 11260 (Section 2.2.4.2). The former is a modification of the method according to Mehlich (1938) and Mehlich (1942). The CEC of soil is determined using a barium chloride ($BaCl_2$) solution, buffered with triethanolamine at pH 8.1. With the latter method, the effective cation exchange capacity (ECEC; 2.2.4.2) of the soil is determined at the original pH and at a low total ionic strength (about 0.01 mol/L). Additionally, the content of exchangeable sodium (Na), potassium (K), calcium (Ca) and magnesium (Mg) in soil is determined.

In both methods, the soil’s exchange sites are saturated with Ba, either using a $BaCl_2$ solution buffered at pH 8.1 (Section 2.2.4.1) or unbuffered solution (Section 2.2.4.2). Subsequently, Ba is replaced and precipitated by the addition of magnesium sulphate ($MgSO_4$). The potential CEC (at pH 8.1) or the effective CEC (ECEC) is determined by analysis of excess Mg in the second, re-exchange solution. Acidified lanthanum solution is used to determine excess magnesium using flame atomic absorption spectrometry (FAAS) in an air/acetylene flame. Lanthanum inhibits the formation of incombustible compounds of magnesium with phosphate, aluminium etc.

Additionally, the sum of the exchangeable cations, i.e. Na, K, Ca and Mg can be quantified in the Ba exchange solution and represents the exchangeable bases (method in 2.2.4.1) or the relative contribution of the bases to the ECEC is termed as the base saturation level (method in 2.2.4.2).

Both methods are applicable to all types of air-dried soil samples; pre-treatment according to ISO 11464 is recommended. A generalised flow chart for both methods is shown in Fig. 2.2.4.1.

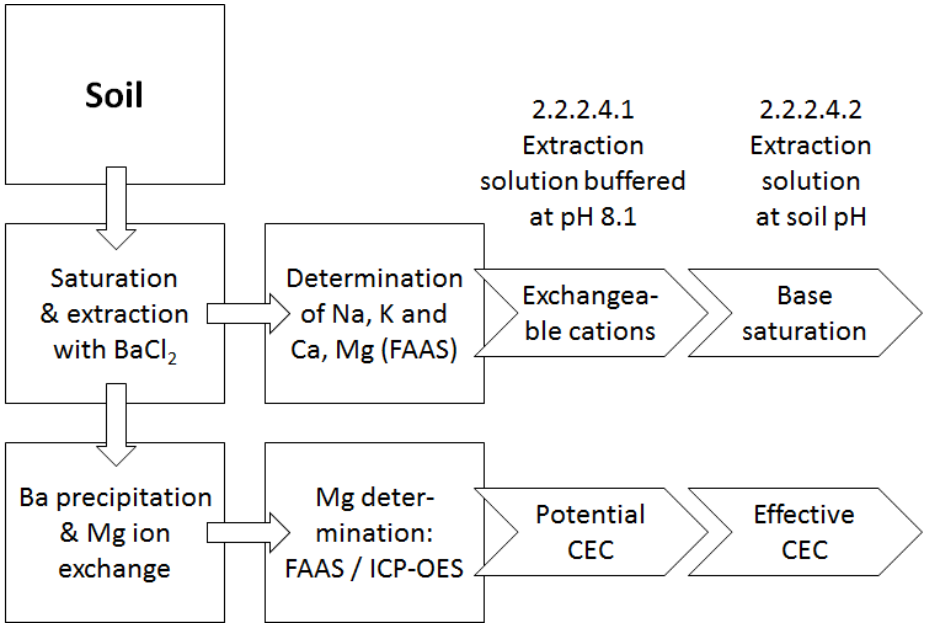

Figure 2.2.4.1 Flow chart of the two-step extraction scheme for the determination of the exchangeable cations and the potential cation exchange capacity and effective cation exchange capacity, respectively

Soil samples are air-dried, sieved <2 mm and additionally microaggregates are carefully destroyed using a mortar and pestle. Soils should not contain higher amounts of soluble salts, calcite and/or gypsum, which is indicated by a higher electric conductivity. Determination of electric conductivity (EC), e.g. using ISO 11265, indicates possible salt affection of soils. Cations released from these compounds will distort the exchangeable cation content. In that case, soil samples must be treated in parallel using water. The exchangeable cation contents determined in this water extract are subtracted from the contents in the $BaCl_2$ extract.

For soils with high sulphate content it might be advisable to determine the CEC with the help of methods using ammonium solutions, e.g. ammonium acetate, instead of Ba.

Materials and equipment

- Shaker, rotary shaker (end-over-end) or horizontal
- Tightly locking polyethylene centrifuge tubes (ca. 50 mL)
- 50 or 100 ml polyethylene (PE) flasks
- Funnels
- Filter paper (Whatman No. 42, Schleicher & Schuell 595 1/2, Macherey-Nagel 261 G 1/4, or similar)
- Glass vacuum line (e.g. electric pump)
- Flame atomic absorption spectrometer (FAAS) or ICP-OES

29.1 Determination of potential CEC and exchangeable cations using a pH 8.1 buffered barium chloride solution

Reagents

Reagents of recognised analytical grade shall be used, including

- Deionised water (electric conductivity < 0.2 mS/m at 25 °C)
- Barium chloride ($BaCl_2$) solution; *c*($BaCl_2$) = 1 mol/L. Preparation: Dissolve 224 g of $BaCl_2 \times 2\ H_2O$ in 1000 mL of water (use volumetric flask)
- Hydrochloric acid, *c*(HCl) = 2 mol/L. Preparation: Dissolve 166 mL of concentrated HCl (ρ = 1.19 g/cm³) in 1000 mL of water (use volumetric flask)
- Triethanolamine solution, pH 8.1. Preparation: Dissolve 90 mL triethanolamine in water in a total volume of 1000 mL. Adjust the pH to 8.1 using about 140 to 150 mL of hydrochloric acid (c). Fill up with water to 2 L
- Extraction solution. Add equal volume fractions of solutions b) and d). Protect the solution during storage from contact with CO_2 and/or prepare fresh solution any time when needed
- Magnesium sulphate solution; *c*($MgSO_4$) = 0.020 mol/L. Preparation: Dissolve 4.930 g magnesium sulphate heptahydrate ($MgSO_4 \times 7\ H_2O$) in 1000 mL of water (use volumetric flask). Prepare a fresh solution. Magnesium sulphate can lose crystal water during storage. Protect from that by wrapping the flask in an additional PE bag and storing the chemical in a refrigerator
- Hydrochloric acid, *c*(HCl) = 12 mol/L (ρ = 1.19 g/cm³)
- Magnesium standard solution; *c*($MgSO_4$) = 0.0010 mol/L. Preparation: Add 50 mL of magnesium sulphate solution f) in a 1000 mL volumetric flask and fill up with water to 1 L. *See Remarks*
- Acidified lanthanum solution: *c*(La) = 10 g/L. Preparation: Add 15.6 g lanthanum nitrate hexahydrate [$La(NO_3)_3 \times 6\ H_2O$] in a 500 mL volumetric flask, add 42 mL hydrochloric acid g), and fill up with water to 500 mL
- Sodium and potassium stock solution: *c*(Na) = 400 mg/L, *c*(K) = 1000 mg/L. Dissolve 1.0168 g sodium chloride and 1.9068 g potassium chloride in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*
- Diluted stock solution: *c*(Na) = 40 mg/L, *c*(K) = 100 mg/L. Pipette 25 mL of solution j) in 250 mL volumetric flask and fill up with water to the mark. *See Remarks*
- Hydrochloric acid, *c*(HCl) = 1 mol/L. Add 83 mL of concentrated HCl (ρ = 1.19 g/cm³) to water, to receive a total volume of 1000 mL
- Calcium stock solution: *c*(Ca) = 1000 mg/L. Dissolve 2.497 g calcium carbonate in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*
- Magnesium stock solution: *c*(Mg) = 100 mg/L. Dissolve 1.0168 g sodium chloride and 0.836 g magnesium chloride hexahydrate ($MgCl_2 \times 6\ H_2O$) in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*

Procedure

Extraction:

a. Weigh air-dried soil in a tightly locking polyethylene centrifuge tube (ca. 50 mL). Use 2.50 g of clayey and/or humus rich soil and 5.00 g of sandy and/or humus poor soil. Note down the total weight of soil plus centrifuge tube (m_1)
b. Add 30 mL of extraction solution e) and shake for 1 h. Centrifuge for 10 min at 3000 g
c. Decant the supernatant into a 100 mL volumetric flask. Repeat this procedure (extraction, shaking, centrifugation, decanting) twice more. Collect all three supernatants in the same volumetric flask. Finally, fill up to the mark with fresh extraction solution
d. Shake well before filtering the whole solution. Save filtrate I for the analysis of the exchangeable bases (Na, K, Ca, Mg)
e. Add 40 mL of water to the precipitate in the centrifugation tube and shake for 1 to 2 min to resuspend. Centrifuge for 10 min at 3000 g. Decant and discard the supernatant
f. Weigh the centrifuge tube together with the remaining content (m_2). Add 30 mL of $MgSO_4$ solution f) and shake overnight. Decant the solution and filter into PE bottles. Store filtrate II for analysis of excess magnesium
g. Prepare blank samples in parallel

Determination of CEC:

a. Pipette 0.20 mL from the filtrates II of the samples and blank samples into 100 mL volumetric flasks. Add 10 mL of acidified lanthanum solution i), fill up with water to the mark and mix. Determine the concentration of magnesium using the diluted filtrates II
b. For calibration, use dilutions of the magnesium standard solution h). Pipette 0 mL, 1 mL, 2 mL, 3 mL, 4 mL, and 5 mL into a series of 100 mL volumetric flasks. Add 10 mL of acidified lanthanum solution to each flask. Final concentration of the calibration solutions: 0, 0.01, 0.02, 0.03, 0.04, and 0.05 mmol/L, respectively
c. Analyse magnesium using FAAS (or with ICP-OES) at a wavelength of 285.2 nm. Use instrumentation settings following the manufacturer's instructions for optimum performance of the instrument

Calculation

The magnesium concentration is measured in filtrate II. This must be corrected for the liquid remaining after decanting in the soil pellet:

$$c_2 = [c_1 \times (30 + m_2 - m_1)] \times 30^{-1} \qquad \text{(Eq. 2.2.4.1.1)}$$

where

$\boldsymbol{c}_1$ is the magnesium concentration in the diluted filtrate II [mmol L^{-1}],

$\boldsymbol{c}_2$ is the corrected magnesium concentration in the diluted filtrate II [mmol L^{-1}],

$\boldsymbol{m}_1$ is the mass of the centrifuge tube plus air-dried soil [g],

$\boldsymbol{m}_2$ is the mass of the centrifuge tube plus moist soil [g].

The cation exchange capacity (CEC) of the soil is calculated with the following equation:

$$CEC = 3000 \times (c_{b1} - c_2)] \times m^{-1}\ [cmol_c\ kg^{-1}] \qquad \text{(Eq. 2.2.4.1.2)}$$

where

c_2 is the corrected magnesium concentration in the diluted filtrate II [mmol L^{-1}],

c_{b1} is the magnesium concentration in the diluted filtrate II of the blank sample [mmol L^{-1}],

m is the mass of the air-dried soil sample [g].

If the CEC exceeds 40 $cmol_c\ kg^{-1}$, the determination should be repeated with less weight of soil sample taken. Adjust all calculations appropriately.

Determination of the exchangeable sodium and potassium:

a. Determine sodium (Na) and potassium (K) in acidified barium chloride/triethanolamine extract of the soil samples using FAAS
b. *Calibration:* Prepare solutions with 0 mL, 5 mL, 10 mL, 15 mL, 20 mL and 25 mL of the diluted stock solution k) in 50 mL volumetric flasks. Add 10.0 mL of extraction solution e) and 5.0 mL of hydrochloric acid l). Fill up to the mark with water. The resulting concentrations of Na are 0, 4, 8, 12, 16, 20 mg/L. The resulting concentrations of K are 0, 10, 20, 30, 40, 50 mg/L
c. *Analysis:* Fill 2.0 mL of the filtrate I and of the blank sample, respectively, into reaction tubes. Add 1.0 mL of hydrochloric acid l) and 7.0 mL of water and mix (*see also Remarks*). Determine Na and K with FAAS (or ICP-OES) at wavelength 589 nm and 766 nm, respectively, with the instrument set according to the manufacturer's instructions for optimum performance
d. *Calculations:* The content of exchangeable Na and K in soil samples is calculated as follows:

$$c(\text{Na, exchangeable}) = 2.1749 \times (c_{sample} - c_{blank}) / m \quad [cmol_c\ kg^{-1}] \qquad \text{(Eq. 2.2.4.1.3)}$$

$$c(\text{K, exchangeable}) = 1.2788 \times (c_{sample} - c_{blank}) / m \quad [cmol_c\ kg^{-1}] \qquad \text{(Eq. 2.2.4.1.4)}$$

with the measured concentrations in the diluted sample (c_{sample}) and the diluted blank sample (c_{blank}), respectively, and m is the soil mass in g.

Determination of the exchangeable calcium and magnesium:

a. Determine calcium (Ca) and magnesium (Mg) in acidified barium chloride/triethanolamine extract of soil samples using FAAS
b. *Calibration*: Prepare solutions with 0 mL, 2 mL, 4 mL, 6 mL, 8 mL and 10 mL of the mixed stock solution k) in 100 mL volumetric flasks. Add 10.0 mL of extraction solution e) and 10.0 mL of hydrochloric acid l). Fill up to the mark with water. The resulting concentrations of Ca are 0, 1, 2, 3, 4, 5 mg L^{-1}. The resulting concentrations of Mg are 0, 0.1, 0.2, 0.3, 0.4, 0.5 mg L^{-1}
c. *Analysis*: Fill 1.0 mL of the filtrate I and of the blank sample, respectively, into reaction tubes. Add 1.0 mL of hydrochloric acid l) and 8.0 mL of water and mix (*see also above comment*). Determine Ca and Mg with

FAAS at wavelength 422.7 nm and 285.2 nm, respectively, with the instrument set according to the manufacturer's instructions for optimum performance

d. *Calculations*: The content of exchangeable Ca and Mg in soil samples is calculated as follows:

$$c(\text{Ca, exchangeable}) = 8.2288 \times (c_{sample} - c_{blank}) / m \quad [\text{cmol}_c\ \text{kg}^{-1}] \text{ (Eq. 2.2.4.1.5)}$$

$$c(\text{Mg, exchangeable}) = 4.9903 \times (c_{sample} - c_{blank}) / m \quad [\text{cmol}_c\ \text{kg}^{-1}] \text{ (Eq. 2.2.4.1.6)}$$

with the measured concentrations in the diluted sample (c_{sample}) and the diluted blank sample (c_{blank}), respectively, and m is the soil mass in g.

29.2 Determination of the effective cation exchange capacity and base saturation level using barium chloride solution

Reagents

Reagents of recognised analytical grade shall be used, including:

- Deionised water (electric conductivity < 0.2 mS m^{-1} at 25 °C)
- Barium chloride ($BaCl_2$) solution; $c(BaCl_2)$ = 0.1 mol L^{-1}. Preparation: Dissolve 24.43 g of $BaCl_2 \times 2\ H_2O$ in 1000 mL of water (use volumetric flask)
- $BaCl_2$ solution; $c(BaCl_2)$ = 0.0025 mol L^{-1}. Preparation: Dilute 25 mL of solution B) in 1000 mL of water
- Magnesium sulphate solution; $c(MgSO_4)$ = 0.020 mol L^{-1}. Preparation: Dissolve 4.930 g magnesium sulphate heptahydrate ($MgSO_4 \times 7\ H_2O$) in 1000 mL of water (use volumetric flask). Prepare fresh solution. Magnesium sulphate can lose crystal water during storage. Protect from that by wrapping the flask in an additional PE bag and storing the chemical in a refrigerator
- Hydrochloric acid, $c(HCl)$ = 12 mol L^{-1} (ρ = 1.19 g cm^{-3})
- Magnesium standard solution; $c(Mg)$ = 0.0010 mol L^{-1}. Preparation: Add 50 mL of $MgSO_4$ solution D) to a 1000 mL volumetric flask and fill up with water to the mark. *See Remarks.*
- Acidified lanthanum solution: $c(La)$ = 10 mg L^{-1}. Preparation: Add 15.6 mg lanthanum nitrate hexahydrate [$La(NO_3)_3 \times 6\ H_2O$] to a 500 mL volumetric flask, add 42 mL hydrochloric acid g), and fill up with water to 500 mL
- Acidified caesium chloride solution: Dissolve 10 g caesium chloride in some water. Add 83 mL of hydrochloric acid E) and make up to 1000 mL with water
- Sodium and potassium stock solution: $c(Na)$ = 400 mg/L, $c(K)$ = 1000 mg/L. Dissolve 1.0168 g sodium chloride and 1.9068 g potassium chloride in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*
- Diluted stock solution: $c(Na)$ = 40 mg L^{-1}, $c(K)$ = 100 mg L^{-1}. Pipette 25 mL of solution j) in 250 mL volumetric flask and fill up with water to the mark. *See Remarks*
- Hydrochloric acid, $c(HCl)$ = 4 mol L^{-1}. Add 83 mL of concentrated HCl (ρ = 1.19 g/cm^3) to water, to receive a total volume of 1000 mL
- Calcium stock solution: $c(Ca)$ = 1000 mg L^{-1}. Dissolve 2.497 g calcium carbonate in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*

- Magnesium stock solution: *c*(Mg) = 100 mg L^{-1}. Dissolve 1.0168 g sodium chloride and 0.836 g magnesium chloride hexahydrate ($MgCl_2 \times 6\ H_2O$) in water. Transfer to 1000 mL volumetric flask and fill up to the mark with water. *See Remarks*

Procedure

Leaching:

a. Weigh 2.50 g of air-dried soil, for example, into a polyethylene centrifuge tube of about 50 ml capacity. Close cap tightly. Note the combined mass of tube and soil (m_1)
b. Add 30 ml of $BaCl_2$ solution B) and shake for 1 h. Subsequently centrifuge the tubes at 3,000 g for 10 min. Note: Balance tubes before centrifugation. Transfer the supernatant liquid to a 100 ml volumetric flask. Repeat this procedure, i.e. the addition of 30 ml of $BaCl_2$ solution, shaking and centrifugation twice more. Collect all three supernatants in the same volumetric flask. Make up to the volume of the volumetric flask with $BaCl_2$ solution B). Mix, filter and store the **extract I** for the determination of the exchangeable concentration of **Na, K, Ca and Mg**. *See Remarks*

Cleansing:

c. Add 30 ml of $BaCl_2$ solution C) to the soil pellet and shake overnight. (Resulting Ba concentration in the equilibrium solution will be about 0.01 mol L^{-1}). Centrifuge tubes at 3,000 g for 10 min. Decant the supernatant liquid

Re-exchange:

d. Weigh the tube with its contents and cap (m_2). Add 30 ml of $MgSO_4$ solution D) to the soil pellet and shake overnight. Centrifuge tubes at 3,000 g for 10 min. Decant the supernatant through a filter paper into a new flask and store the **extract II** for the determination of the concentration of **excess magnesium** (see below)
e. Prepare blank samples without the addition of soil in parallel and follow the above described procedure completely

Determination of CEC:

a. Pipette 0.20 mL from the extracts II of the samples and blank samples into 100 mL volumetric flasks
b. Add 0.3 mL of the $BaCl_2$ solution B) and additional 10 mL of acidified lanthanum solution G).
c. Fill up with water to the mark and mix
d. For calibration, use dilutions of the magnesium standard solution F). Pipette 0 mL, 1 mL, 2 mL, 3 mL, 4 mL, and 5 mL into a series of 100 mL volumetric flasks. Add 10 mL of acidified lanthanum solution G) to each flask and fill up to the mark with water. Final concentration of the calibration solutions: 0, 0.01, 0.02, 0.03, 0.04, and 0.05 mmol/L, respectively
e. Analyse magnesium using FAAS at a wavelength of 285.2 nm (or ICP-OES) with instrumentation settings following the manufacturer's instructions for optimum performance of the instrument

Calculation

Correct the concentrations of magnesium in the sample solutions for the volume of the liquid retained by the centrifuged soil after being treated with 0,0025 mol L^{-1} $BaCl_2$ solution:

$$c_2 = [c_1 (30 + m_2 - m_1)] / 30 \qquad \text{(Eq. 2.2.4.2.1)}$$

where

c_2 is the corrected magnesium concentration in the sample [mmol / L],

c_1 is the magnesium concentration in the sample [mmol / L],

m_1 is the mass of the centrifuge tube with air-dried soil [g],

m_2 is the mass of the centrifuge tube with wet soil [g].

Calculate the cation exchange capacity (CEC) of the soil using the following equation:

$$CEC = (c_{b1} - c_2)\ 3{,}000 / m \qquad \text{(Eq. 2.2.4.2.2)}$$

where

CEC is the cation exchange capacity of the soil [$cmol_c\ kg^{-1}$],

c_2 is the corrected magnesium concentration in the sample [$mmol\ L^{-1}$],

c_{b1} is the magnesium concentration in the blank [$mmol\ L^{-1}$],

m is the mass of the air-dried sample [g].

If the CEC exceeds 40 $cmol_c\ kg^{-1}$, the determination should be repeated using less soil, adjusting the calculation accordingly.

Determination of the exchangeable sodium and potassium

a. Determine sodium (Na) and potassium (K) in acidified $BaCl_2$ extract of soil samples using FAAS. To eliminate ionisation interference, a caesium solution is added to the samples
b. *Calibration:* Prepare solutions with 0 mL, 5 mL, 10 mL, 15 mL, 20 mL and 25 mL of the diluted stock solution J) in 50 mL volumetric flasks. Add 10.0 mL of extraction solution B) and 5.0 mL of acidified caesium chloride solution H). Fill up to the mark with water. The resulting concentrations of Na are 0, 4, 8, 12, 16, 20 mg/L. The resulting concentrations of K are 0, 10, 20, 30, 40, 50 mg L^{-1}. *See Remarks*
c. *Analysis:* Fill 2.0 mL of the extract I and of the blank sample, respectively, into reaction tubes. Add 1.0 mL of acidified caesium chloride solution H) and 7.0 mL of water and mix (*See Remarks*). Determine Na and K with FAAS at wavelength 589 nm and 766 nm, respectively, with the instrument set according to the manufacturer's instructions for optimum performance
d. *Calculations:* The content of exchangeable Na and K in soil samples is calculated as follows:

$$c(\text{Na, exchangeable}) = 2.1749 \times (c_{sample} - c_{blank}) / m \quad [cmol_c\ kg^{-1}] \qquad \text{(Eq. 2.2.4.2.3)}$$

$$c(\text{K, exchangeable}) = 1.2788 \times (c_{sample} - c_{blank}) / m \quad [cmol_c\ kg^{-1}] \qquad \text{(Eq. 2.2.4.2.4)}$$

with the measured concentrations in the diluted sample (c_{sample}) and the diluted blank sample (c_{blank}), respectively, and m is the soil mass in g.

Determination of the exchangeable calcium and magnesium

a. Determine calcium (Ca) and magnesium (Mg) in acidified barium chloride/triethanolamine extract of soil samples using FAAS
b. Calibration: Prepare solutions with 0 mL, 2 mL, 4 mL, 6 mL, 8 mL and 10 mL of the mixed stock solution J) in 100 mL volumetric flasks. Add 10.0 mL of extraction solution B) and 10.0 mL of hydrochloric acid K). Fill up to the mark with water. The resulting concentrations of Ca are 0, 1, 2, 3, 4, 5 mg L^{-1}. The resulting concentrations of Mg are 0, 0.1, 0.2, 0.3, 0.4, 0.5 mg/L. *See Remarks*
c. Analysis: Fill 1.0 mL of the filtrate I and of the blank sample, respectively, into reaction tubes. Add 1.0 mL of hydrochloric acid l) and 8.0 mL of water and mix (*see also above comment*). Determine Ca and Mg with FAAS at wavelength 422.7 nm and 285.2 nm, respectively, with the instrument set according to the manufacturer's instructions for optimum performance
d. Calculations: The content of exchangeable Ca and Mg in soil samples is calculated as follows:

$$c(\text{Ca, exchangeable}) = 8.2288 \times (c_{sample} - c_{blank}) / m \quad [\text{cmol}_c\ \text{kg}^{-1}] \qquad \text{(Eq. 2.2.4.2.5)}$$

$$c(\text{Mg, exchangeable}) = 4.9903 \times (c_{sample} - c_{blank}) / m \quad [\text{cmol}_c\ \text{kg}^{-1}] \qquad \text{(Eq. 2.2.4.2.6)}$$

with the measured concentrations in the diluted sample (c_{sample}) and the diluted blank sample (c_{blank}), respectively, and m is the soil mass in g.

Examples

Some examples for values of potential and effective cation exchange capacity are given in Table 2.2.2.4.1. It can be seen that with decreasing soil pH, the base saturation decreases and the difference between potential CEC and ECEC increases. While exchange sites in soils with pH 6.5 and higher are largely dominated by Ca, Al is the dominant cation covering exchange sites in acidic soils

Table 2.2.4.2.1 Typical values of data on potential CEC, effective CEC, saturation with acidic (Al, Mn, Fe) and basic (Na, K, Ca, Mg) cations and base saturation (Base sat.) in soils of different climates and soil use. Data from Blume et al., (2010) and own data

Soil	Parent rock	pH	OC	pot. CEC	ECEC	Saturation %							Base sat.
		$CaCl_2$	%	$cmol_c\ kg^{-1}$		Al	Mn	Fe	Na	K	Ca	Mg	%
Arable soils (central Europe)													
Luvisol	loess	6.3	1.4	17	14	<LOD[a]	–[b]	–	<1	5	80	15	100
Chernozem	loess	7.2	1.6	18	18	<LOD	–	–	0.4	0.5	90	9	100
Vertisol	mudstone	6.7	2.4	22	17	<LOD	–	–	<LOD	9	83	8	100
Cambisol	terrace mat.	6.6	1.6	13	11	<LOD	0.7	0.02	0.5	3.3	77	20	100
Cambisol	clay stone	6.6	1.9	14	11	<LOD	0.7	0.08	0.3	7.2	73	20	100

Forest soils (central Europe)													
Podzol	granite	2.6	12	17	6.8	65	–	–	2.0	5	22	6	35
Stagnosol	loess	3.8	5.7	18	5.4	69	–	–	11	6	13	<2	30
Cambisol	loess	2.9	20	60	12	85	–	–	<LOD	5	5.8	4.2	15
Soils in other climates													
Vertisol		6.8	0.9	45	47	<LOD	–	–	3.6	0.4	71	25	100
Ferralsol		3.5	2.8	13	2.6	89	–	–	1.2	3.1	2.7	3.5	11
Acrisol		3.5	3.3	26	7.2	72	–	–	1.4	2.8	15	8.3	28

[a] <LOD = below limit of detection; [b] – = parameter was not measured

Remarks

- If the barium chloride extract has a yellowish-brown colour, this indicates that some organic matter has been dissolved. If this occurs, record it in the test report
- As an alternative to the preparation of standards and calibration series, respectively, certified standard solutions are commercially available; aliquots are diluted as required
- For a complete analysis of exchangeable cations, it might be reasonable to additionally determine NH_4^+ in fertilised agricultural soil and exchangeable Al in acidic and also Fe in very acidic soil
- Any other volumes can be used as well, as long as the same concentrations are obtained and the final sample volume is sufficient for analysis with FAAS or ICP-OES
- Dilutions can be prepared much faster doing pipette dilutions and using a diluter system, respectively
- Note: The unit cmolc/kg replaces the old unit millli-equivalents/100 g
- There is a contradiction between guidelines ISO 13536 and ISO 11260. In the former an acidified lanthanum solution containing 10 g L^{-1} must be prepared, while here the concentration of La is 10 mg L^{-1}. Typically, a La concentration of 10 g L^{-1} is recommended
- Analysis of sodium and potassium for ECEC is supposed to be done with acidified caesium chloride solution (Section 2.2.4.2), while for potential CEC this is done with hydrochloric acid (Section 2.2.4.1). The user can decide whether to employ acidified caesium chloride or not

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30. Total nutrients and metals (Ca, Mg, K, Fe, Mn, Cu, Zn, Cd, Pb, Ni, Cr, As, Al, B)

José A. Acosta, Silvia Martínez-Martínez, Raúl Zornoza, Virginia Sánchez-Navarro, Ángel Faz

Sustainable Use, Management, and Reclamation of Soil and Water Research Group,

Universidad Politécnica de Cartagena, Paseo Alfonso XIII, 48, 30203, Cartagena, Spain.

Importance and applications

All plants require 17 elements to complete their life cycle, and an additional four elements have been identified as essential for some plants (Havlin et al., 2005). With the exception of C, H, and O, which plants obtain from air and water, plants derive the remaining 14 elements from the soil or through fertilisers, manures, and amendments (Parikh & James, 2012). The bulk of the soil solid fraction is constituted by soil minerals, which exert significant direct and indirect influences on the supply and availability of most nutrient elements. Soil parent material has a significant direct influence on the nutrient element contents of the soil, and on their concentrations depending on rock type. Therefore, in order to better understand the dynamic of nutrients in soil, it is useful to determine their total concentrations.

In addition, soils may become contaminated by the accumulation of heavy metals and metalloids through anthropogenic activities. The adequate protection and restoration of soil agro-ecosystems contaminated by heavy metals requires a detailed soil characterisation, with total metals concentrations being an essential parameter.

Principle

A representative sample is extracted and/or dissolved in concentrated nitric acid, or alternatively, concentrated nitric acid and concentrated hydrochloric acid using microwave heating with a suitable laboratory microwave unit. The sample and acid(s) are placed in a microwave vessel. The vessel is sealed and heated in the microwave unit for a specified period of time. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analysed using the appropriate determinative method (USEPA, 1997).

Reagents

- Concentrated nitric acid (HNO_3) 65%
- Concentrated hydrochloric acid (HCl) 37%
- Deionised water

Materials and equipment

- Volumetric flask (100 mL)
- Funnels
- Quantitative filter papers of 110 mm diameter (0.45 □m pore size)
- Pipette (10 mL)
- Microwave unit
- Vessels
- Analytical balance

- Flame atomic absorption spectrophotometer (FLAA) or graphite furnace atomic absorption spectrophotometer (GFAA) or inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS)

Procedure

a. Weigh 0.500 g of well-mixed ground soil sample into a microwave vessel to the nearest 0.001 g with an appropriate analytical balance
b. Add 10 mL concentrated nitric acid or, alternatively, 9 mL concentrated nitric acid and 3 mL concentrated hydrochloric acid to the vessel in a fume hood. The addition of concentrated hydrochloric acid to the nitric acid is appropriate for the stabilisation of high Fe and Al concentrations in solution
c. Seal the vessel according to the manufacturer's instructions. Properly place the vessel in the microwave system according to the manufacturer's recommended specifications and, when applicable, connect appropriate temperature and pressure sensors to vessels according to the manufacturer's specifications
d. Start the microwave program. The temperature of each sample should rise to 175 ± 5ºC in approximately 5.5 ± 0.25 min and remain at 175 ± 5ºC for 4.5 min, and stay for at least 10-min reducing the temperature (Fig. 2.2.5.1)
e. At the end of the microwave program, allow the vessels to cool for a minimum of 30 min before removing them from the microwave system. Cooling of the vessels may be accelerated by internal or external cooling devices
f. Complete the preparation of the sample by venting microwave containers in a fume hood before uncapping, so as to avoid a rush of acid vapour that may still be in the headspace. When cool enough to handle, carefully uncap the vessels
g. Filter the sample solution through quantitative filter paper into a 100 mL volumetric flask, and make up to 100 mL with deionised water
h. The solution is now ready for analysis for elements of interest using appropriate elemental analysis techniques (Flame atomic absorption spectrophotometer (FLAA) or graphite furnace atomic absorption spectrophotometer (GFAA) or inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS)

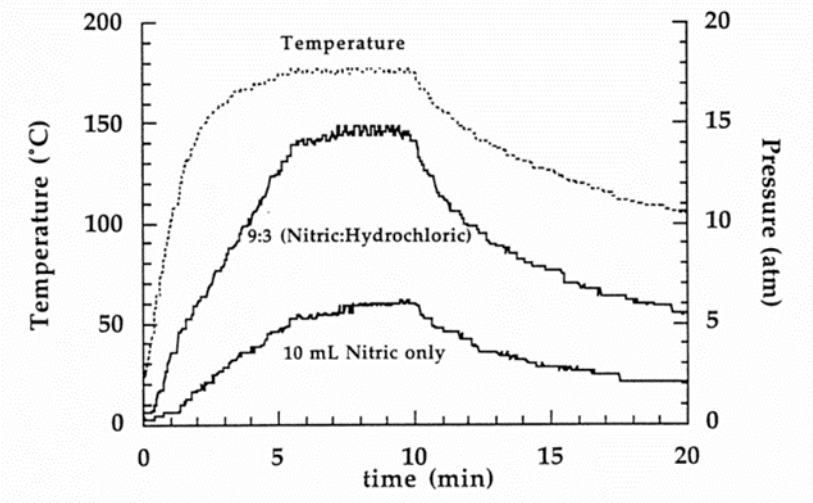

Figure 2.2.5.1 Temperature and pressure profile (Link et al., 1997, 1998)

Calculations

Convert the extract concentration obtained from the instrument in mg/L to mg/kg dry-weight of sample by

$$\text{Sample concentration (mg kg}^{-1}) = \frac{\text{X V}}{\text{W}} DF \qquad \text{(Eq. 2.2.5.1)}$$

where

X is the concentration obtained from the instrument [mg L^{-1}],
V is the final volume of the sample solution [mL] (e.g. volumetric flask of 100 mL),
W is the weight of the sample [g],
DF is the dilution factor (DF = 1.00 with no sample dilution).

Remarks

- All digestion vessels must be carefully acid washed and rinsed with reagent water. When switching between high concentration samples and low concentration samples, all digestion vessels should be cleaned by leaching with hot (1:1) hydrochloric acid (greater than 80ºC, but less than boiling) for a minimum of two hours followed by hot (1:1) nitric acid (greater than 80ºC, but less than boiling) for a minimum of two hours. The vessels should then be rinsed with deionised water and dried in a clean environment
- The addition of hydrochloric acid may limit the quantitation techniques and increase the difficulties of analysis for some quantitation systems

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31. Bioavailable nutrients and metals (Fe, Mn, Cu, Zn, Cd, Pb, Ni, Cr, As, Al)

José A. Acosta, Silvia Martínez-Martínez, Raúl Zornoza, Virginia Sánchez-Navarro, Ángel Faz

Sustainable Use, Management, and Reclamation of Soil and Water Research Group,

Universidad Politécnica de Cartagena, Paseo Alfonso XIII, 48, 30203, Cartagena, Spain.

Importance and applications

Metals are associated to different fractions in the soil: (1) in soil solution, as free metal ions and soluble metal complexes, (2) adsorbed in the exchange sites of the soil inorganic constituents, (3) bound to organic matter, (4) precipitated as oxides, hydroxides and carbonates, and (5) in the structures of silicate minerals (Rieuwerts et al., 1998, Lassat, 2001, Reichman, 2002, Basta, 2004). The bioavailability is defined as the heavy metal fraction available for plant uptake. For sustainable farming, the bioavailable nutrients and metals must be monitored to ensure the necessary amount of nutrients for high quality and optimal production, and to be sure that the concentrations of toxic elements (e.g. lead, cadmium, arsenic etc.) remain below the limits of toxicity for crops. In addition, the monitoring of nutrients allows us to optimise the application of the necessary amount of those elements in order to avoid over-application, which could contaminate the soil, subsoil and even groundwater and increase production costs. Finally, the monitoring of potential toxic metals allows to evaluate the risk of transfer to the food chain, and the need to use techniques for reducing their availability which will minimise this risk.

The bioavailability depends on the metals solubility and their adsorption capacity in the colloidal fraction of soil. The interaction between the different processes such as cation exchange, adsorption/desorption, precipitation/dissolution and complex formation affect the distribution of metals between the soil solution and the solid phase, being responsible for their mobility and bioavailability (Rieuwerts et al., 1998). In addition, soil properties and constituents affect metals bioavailability, such as the pH, redox potential, texture, content and type of clays, organic matter, Fe, Mn and Al oxides, and the presence of cations and anions in solution (Rieuwerts et al., 1998, Reichman, 2002, Silveira et al., 2003; Basta, 2004). In order to determine the concentration of bioavailable metals, chelating agents have been widely used, such as is the case of EDTA and DTPA (Kabata-Pendias, 2000, Reichman, 2002).

Principle

A representative sample is extracted by a chelating agent, DTPA or EDTA. The sample and chelating agent are placed in a plastic container and shaken for a specified period of time. After that, the vessel contents are centrifuged, or allowed to settle, and filtered, then analysed by the appropriate determinative method.

Reagents

Soils with pH >6: DTPA 0.05 M at pH 7.30 solution (Lindsay & Norvell, 1978; Crock & Severson, 1980):

- DTPA (diethylene-triamine-pentaacetic acid)
- $CaCl_2.2H_2O$ (0,01 N)
- Triethanolamine (TEA) (0.1 M)
- HCl (37%).

Soils with pH < 6: EDTA 0.005 M at pH 4.65 solution (Lindsay & Norvell, 1978, Borggaard, 1976):

- EDTA (ethylene-diamine-tetraacetic acid)
- Ammonium acetate ($AcNH_4$)
- HCl (37%)

Materials and equipment

- Beakers (250 and 1000 mL)
- Centrifuge tubes (50 mL)
- Funnels
- Plastic container (50 mL)
- Quantitative filter papers of 110 mm diameter (0.45 □m pore size)
- Analytical balance
- Centrifuge unit
- Orbital shaker
- Flame atomic absorption spectrophotometer (FLAA) or graphite furnace atomic absorption spectrophotometer (GFAA) or inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS)

Procedure

Soil with pH > 6: DTPA 0.05 M at pH 7.30:

a. Preparation of DTPA solution:
   - Weigh the following reagents into a 250 mL beaker:
     - DTPA (diethyene-triamine-pentaacetic acid): 1.9667 g
     - CaCl2.2H2O (0,01 N): 0,0735 g
     - Triethanolamine (TEA) (0.1 M): 14 mL (TEA 98 %) or 15.6 mL (TEA 85 %).
   - Shake the solution.
   - Make up to 1 L with deionised water.
   - Measure the initial pH of the solution and adjust it to 7.3 by gradually adding HCl (37%).

b. Weigh 15 g of well-mixed 2 mm-sieved soil sample into a centrifuge tube to the nearest 0.001 g with an appropriate analytical balance.

c. Add 30 mL of DTPA solution (1:2 ratio soil/solution)

d. Shake for 2 h in an orbital shaker unit.

e. Centrifuge the tube at 2100 rpm for 5 min.

f. Filter the sample solution through quantitative filter paper into a 50 mL container.

g. Flame atomic absorption spectrophotometer (FLAA) or graphite furnace atomic absorption spectrophotometer (GFAA) or inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS).

Soil with pH < 6: EDTA 0.005 M at pH 4.65:

a. Preparation of EDTA solution:
   - Weigh the following reagents into a 250 mL beaker:
     - EDTA (ethylene-diamine-tetraacetic acid): 1.8612 g.
     - Ammonium acetate (AcNH4): 77 g.
   - Shake the solution.

- Make up to 1 L with deionised water.
- Measure the initial pH of the solution and adjust it to 4.65 by gradually adding HCl (37%).

b. Weigh 8 g of well-mixed 2 mm-sieved soil sample into a centrifuge tube to the nearest 0.001 g with an appropriate analytical balance.
c. Add 40 mL of EDTA solution (1:5 ratio soil/solution)
d. Shake for 1 h in an orbital shaker unit.
e. Centrifuge the tube at 2100 rpm for 5 min.
f. Filter the sample solution through quantitative filter paper into a 50 mL container.
g. Flame atomic absorption spectrophotometer (FLAA) or graphite furnace atomic absorption spectrophotometer (GFAA) or inductively coupled plasma atomic emission spectrometer (ICP-AES) or inductively coupled plasma mass spectrometer (ICP-MS).

Calculations

- Convert the extract concentration obtained from the instrument in mg/L to mg/kg dry-weight of sample by:

$$\text{Sample concentration } (\text{mg kg}^{-1}) = \frac{\text{X V}}{\text{W}} DF \qquad \text{(Eq. 2.2.6.1)}$$

where

X is the concentration obtained from the instrument [mg L^{-1}],
V is the final volume of the sample solution [mL],
W is the weight of the sample [g],
DF is the dilution factor (DF = 1.00 with no sample dilution).

Remarks

- In order to choose the correct chelating agent, soil pH must be determined.

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32. Total carbon (organic and inorganic carbon) and nitrogen

Elvira Díaz Pereira, María Martinez-Mena, Joris de Vente, María Almagro Bonmatí, Carolina Boix-Fayos

Soil and Water Conservation Research Group. CEBAS-CSIC.

Campus Universitario de Espinardo, 30100, Murcia, Spain

Importance and applications

Organic Carbon (OC) is the main source of energy and nutrients for soil microorganisms, affecting plant growth. It plays a crucial role in aggregate stability and consequently intervenes in the distribution of the porous space, water holding capacity, and soil moisture, amongst other soil properties. Total Organic Carbon (TOC) affects most of the chemical, physical and biological soil properties linked to their quality, sustainability and productive capacity. An increase in Soil Organic Matter (SOM), and therefore total carbon (C), leads to greater biological diversity in the soil, thus increasing biological control of plant diseases and pests. There are management practices that cause a detriment of TOC over time, while there are practices that facilitate its accumulation. The scientific literature points out that conventional agricultural land management, with intensive tillage, promotes the release of C into the atmosphere, while conservation agriculture favours the accumulation of C in organic forms within the soil (Almagro et al., 2016).

Total nitrogen (TN) corresponds to ammonium-N, nitrate-N, nitrite-N and organic N, around 90–95% of TN in soils is in organic form, and therefore is assimilated by plants through mineralisation. The amount of available nitrogen depends on cultivation methods, the environmental conditions, the expected yield, the nutrients available in the soil and their transformations. The amount of nitrogen needed as fertiliser can be estimated by medium-term analysis of the inputs and outputs of nitrogen forms (balance). A TOC/N ratio of 10–12 indicates a correct release of nitrogen, values above or below provide low or excessive release.

Both TOC and TN are indicators of soil quality, and along with P were recently identified as key biological indicators in relation to land use management across Europe. Both relate to two key soil ecosystem services (carbon cycling and storage potential and nutrient cycling) as detailed by Creamer et al., (2016).

This method is used for the determination of TC and TN by an elemental CN analyser, as well as TOC in soil samples, prior to the elimination of carbonates (if present) with HCl.

Principle

The elementary C and N analyser determines the C and N content of a variety of materials and soils. In this method, C is measured as carbon dioxide from the combustion of the sample by means of an infrared detector. The present N is determined by the Dumas method, by complete combustion in the presence of oxygen, reduction of the oxides of nitrogen formed to molecular nitrogen and its detection with a thermal conductivity detector. The quantification of both elements is carried out with certified reference standards of different concentrations of nitrogen and carbon.

Reagents

- 2N Hydrochloric acid

Materials and equipment

- Elemental Analyzer
- Crucible
- Plastic tips for the different micropipettes
- Micro Spatula
- Tin Capsules
- Stainless Steel Plate
- Heating Plate
- Analytical balance with a precision of 0.0001 g
- Micropipettes of 100 µl
- Laminar flow Hood
- Agate mortar and pestle and ball mill
- Desiccator and silica gel

Procedure

Carbon/total nitrogen:

a. Prepare air-dried soil samples
b. Sieve at 2 mm
c. Grind the sample in a ball mill or agate mortar and pestle
d. Weigh 0.05-0.10 ± 0.01 g of the sample in a tin capsule and then close for later insertion into the Elemental Analyzer
e. Weigh soil calibration standards for known carbon/total nitrogen values

Total organic carbon

a. Prepare air-dried soil samples
b. Sieve at 2 mm
c. Grind the sample in a ball mill or agate mortar and pestle
d. Weigh 0.05–0.07 g of sample in a triple tin capsule
e. Place it on a stainless-steel plate in an orderly manner above a heating plate, at a temperature of about 120ºC.
f. Add 100 µl of 2N HCl repeatedly until the carbonates have been destroyed and the effervescence ceased, and allow the samples to dry for 8 h
g. Close them for later introduction in the Elemental Analyzer
h. Verify that the treatment with HCl has been done correctly using two standards
i. Weigh soil calibration standards for known total organic carbon values

Calculations

The final result is displayed as weight percentage, by multiplying it by 10 it can be expressed in mg g^{-1}.

Table 2.2.7.1 Range of values in different agricultural soils

N (mg g^{-1})	Land use	Soil type	Reference
0.8–1.3	Almond RT vs RTG/NT	Mediterranean climate Calcisols (FAO, 2006)	Martinez-Mena et al., (2013)
1.7–6.1	Cropland vs Grassland	Continental climate (Bavarian soils)	Capriel (2013)
1.0–1.36	Vineyard conventional vs organic	Mediterranean climate Calcareous silty-clay	Coll et al., (2011)
0.5–1.9	Olive conventional vs organic	Mediterranean climate Eutric Cambisols (FAO-ISRICISSS, 2006)	Parras-Alcántara et al., (2015)
TOC (mg g^{-1})	**Land use**	**Soil type**	**Reference**
17.9–26.6	Almond CT vs RT/RTG	Mediterranean climate Calcisols (FAO, 2006)	Almagro et al., (2016)
17–62	Cropland vs Grassland	Continental climate (Bavarian soils)	Capriel (2013)
10.2–13.5	Vineyard conventional vs organic	Mediterranean climate Calcareous silty-clay	Coll et al., (2011)
4.4–22.5	Olive conventional vs organic	Mediterranean climate Eutric Cambisols (FAO-ISRICISSS, 2006)	Parras-Alcántara et al., (2015)

Remarks

- Sample sizes range from 1 to 10 mg
- A large number of samples can be inserted into the loading head simultaneously
- Total analysis takes less than four minutes
- Carbonates can be inferred through the following equation: $CaCO_3$ = (TC-TOC) x 100/12, if TC and TOC are expressed as percentages, then consequently carbonates also, by multiplying it by 10 it can be expressed in mg g^{-1}.

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33. Carbonates

József Dezső and Dénes Lóczy

Institute of Geography and Earth Sciences, Faculty of Sciences, University of Pécs,

7624 Pécs, Ifjúság útja 6.

Importance and applications

The total carbonate content of the soil covers all carbonate ($^{-}CO_3$) minerals. Their formation and importance varies with pedo-climatic regions, but it is their common feature that the soil solution contains carbonic acid.

The *primary source* of carbonates is calcite, dolomite, gypsum, marl and calcareous sandstone. Dolomite and vermiculite are also major sources of magnesium. Carbonated lime accumulates in the function of soil water budget during the weathering of $Ca/Mg(CO_3)_2$ present in carbonate rocks. In many cases, soil subtypes are identified by the distribution of carbonates in the profile. Carbonates buffer soil pH and contribute to the formation of soil structure.

Secondary pedogenic carbonates are precipitation forms. The effectiveness of lime heavily depends on its particle size. Lime grains coarser than 250 microns (0.25 mm) have little value in raising soil pH, at least in the short term. Carbonates in the soil profile appear in the following forms:

- uniformly dispersed, non-visible to the naked eye,
- bound to microchannels, passages, aggregate surfaces, clearly visible;
- in spherical concretions,
- in thick precipitations, horizons, banks.

$CaCO_3$ provides a reactive surface for adsorption and precipitation reactions, for example, of P, trace metals and organic acids (Talibudeen & Arambarri, 1964; Amer et al., 1985). Adequate calcium helps delay leaf senescence and slows down or prevents leaf and fruit fall (abscission). Plants take up calcium in the ionic form (Ca^{2+}).

Carbonate deficit causes ‘blossom-end’ rot in tomatoes. It can be induced by moisture stress, even though the soil may have the adequate calcium levels required for cell elongation and cell division. Deficit is manifested in the chlorosis of young parts, deformation of leaves and browning of leaf veins. In acute cases, root growth stops growth peak gets brown and dies. Wilting may occur even with a good water supply.

Carbonate surplus (higher than 15% carbonate content) leads to phosphorus binding and reduced uptake of microelements (Cu, Zn, Mn, Fe, B).

Principle

When free carbonates are present, the acid will produce effervescence due to the release of CO_2 gas (Loeppert & Suarez, 1996).

The experiments to determine dissolved carbonates in soil samples use Scheibler's calcimeter, a volumetric method (EN ISO 10693:2014). The carbonates present in the sample are converted into CO_2 by adding hydrochloric acid to the sample:

$$CaCO_3 + 2\ HCl = CO_2 + CaCl_2 + H_2O \qquad \text{(Eq. 2.2.8.1)}$$

As a result of the pressure of the CO_2 released, the water in a burette that is de-aerated rises. The difference in level measured is an indication of the released CO_2, from which the carbonate content can be calculated. At 30 seconds, the pressure is recorded as '$CaCO_3$ pressure'. If the test sample contains any dolomite, there will be a pause, then a slow, second rise in pressure. The reaction is complete when the pressure stops growing (within 30 to 45 minutes). The final pressure value is the total $CaCO_3$ pressure plus the dolomite pressure. The carbonate content is expressed as an equivalent calcium carbonate content. The Scheibler apparatus designed for kinetic dissolution of carbonate is shown in Fig. 2.2.8.1.

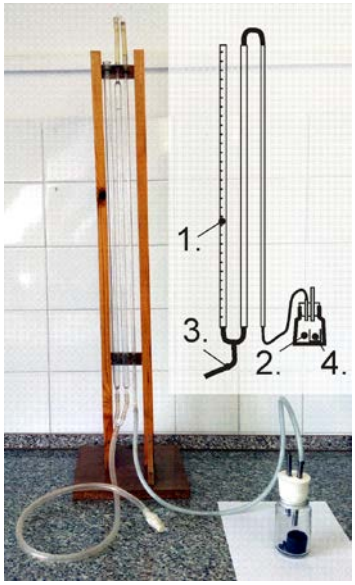

Fig. 2.2.8.1 Scheibler's calcimeter system. 1, U-shaped calibrated manometer with volumetric cm^3 degree, 2, lockup glass reaction vessel tube with a cubic capacity of 100-200 cm^3 connected to the manometer, 3, pipe and tap for regulation of water level, 4, small tubes to hold the acid

Reagents

- 10% HCl
- distilled water

Materials and equipment

- Scheibler apparatus
- Thermometer (accuracy: 0.1°C)
- Barometer (accuracy: 1hPa)

- Scale (accuracy: 0.01g)

Procedure

a. Prepare dried soil sample (at 120°C in oven for 24 h)

b. For the preliminary determination, check the carbonate content of the sample through dripping 10% hydrochloric acid on it (Table 2.2.8.1). Weigh the soil sample into the vessel depending on the intensity of effervescence. Fizzle intensity is shown on a four-grade range

c. Fill a reaction vessel with a soil sample

d. Place the test tube with hydrochloric acid in the reaction vessel using a pair of tweezers (prepare one reaction vessel for each burette). Close the reaction vessel.

e. Fill the manometer up to 0 point with distilled water.

f. Enable the hydrochloric acid to flow out of the test tube and react with the soil sample contains $CaCO_3$, initiating the reaction

g. Swirl the reaction cell and allow sufficient time for the reaction to finish

h. Perform the procedure three times

Table 2.2.8.1 Relative fizzing values and amount of investigated soil sample

Fizzle	Notation	Required soil sample (g)
No fizzle	0	no measurement required
Slight fizzle	x	1.5-2
Medium fizzle	xx	0.5-1.5
Intensive fizzle	xxx	0.2-0.5

Calculations

There are two ways to calculate soil carbonate content (Campbell & Norman, 1998):

1. *Using ideal gas law:*

$$P\ (theoretical)=(n*R*T)/V \quad [Pa] \quad (Eq.\ 2.2.8.2)$$

where

P is the value measured,

n is the pure $CaCO_3$ amount which is used in the analysis [mol],

R is the universal gas constant [8.3144 $J \cdot mol^{-1} \cdot K^{-1}$],

T is the temperature [K],

V is the volume of the CO_2 released during the process [ml].

At standard temperature and pressure (STP: 0°C and 101.325 kPa) the molar density (Greek letter: ρ_m) of any gas is 44.615 mol m^{-3} (and 1 mol of any gas occupies 22.4 dm^3). From the Boyle-Charles law, the molar density of CO_2 (and any gas) can be computed:

$$\rho_m = 44.615\ (p\ 101.325^{-1})\ (273.15\ T^{-1}) \qquad \text{(Eq. 2.2.8.3)}$$

where

44.615 is the molar density at STP [mol m^{-3}],

p is the air pressure [kPa],

T is the actual temperature [K].

2. *Using auxiliary table* (Table 2.2.8.2):

$$w\ (CaCO_3) = (V*f)\ m^{-1} \quad [\%] \qquad \text{(Eq. 2.2.8.4)}$$

where

w is the soil carbonate content [m/m%],

V is the CO_2 volume released during the process [ml],

m is the soil weight [g],

f factor depending on actual air temperature and pressure (from auxiliary table).

Table 2.2.8.2 Auxiliary table. Conversion of the measured CO_2 to carbonate. The numbers express the weight of carbonate in [g x 10^6] (e.g.: 4114 = 0.004114 g) which refer to 1 cm^3 CO_2

Temp. [°C]	Air pressure [Hgmm]									
	749.00	751.00	753.50	756.00	758.00	760.00	762.50	765.00	767.00	769.00
27	4099	4114	4129	4143	4158	4169	4179	4190	4200	4211
26	4114	4139	4144	4158	4172	4183	4193	4204	4214	4225
25	4128	4143	4158	4172	4186	4197	4208	4219	4230	4241
24	4142	4157	4172	4186	4200	4211	4222	4233	4244	4255
23	4156	4171	4186	4200	4214	4226	4237	4248	4259	4270
22	4170	4185	4200	4214	4228	4240	4252	4263	4274	4285
21	4184	4199	4214	4229	4243	4255	4267	4279	4290	4301
20	4199	4214	4229	4243	4257	4269	4281	4292	4303	4214
19	4213	4228	4243	4258	4272	4284	4296	4307	4318	4329
18	4228	4243	4258	4272	4286	4298	4310	4321	4332	4343
17	4242	4257	4272	4296	4300	4312	4324	4335	4346	4357
16	4256	4271	4286	4300	4314	4326	4338	4349	4360	4371
15	4271	4286	4301	4315	4329	4341	4353	4364	4375	4386

To calculate the dolomite pressure, subtract the CaCO3 pressure (30 second reading) from the total pressure (30–45 minute reading).

Table 2.2.8.3 Evaluation of carbonate content

Carbonate (%)	Category
0	Absence
0.1–4.9	Poorly calcareous
5.0–19.9	Moderately calcareous
> 20.0	Strongly calcareous

Remarks

- Equivalent $CaCO_3$ may be overestimated if HCl reacts with non-carbonated substances in the soil.
- Dolomite and magnesite are completely dissolved, but only part of siderite.
- Analysing Ca and Mg in the solution makes it possible to distinguish between $CaCO_3$ and $MgCO_3$.

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34. Soil Organic Carbon. Functional pools

María Martínez-Mena, Elvira Díaz Pereira, Joris de Vente, María Almagro Bonmatí, Carolina Boix-Fayos

Soil and Water Conservation Research Group, CEBAS-CSIC,

Campus Universitario de Espinardo, 30100, Murcia, Spain

Importance and applications

Organic matter and organic matter fractions are important attributes of soil quality (Gregorich et al.,1994). Soil organic matter (SOM) consists of various functional pools that are stabilised by specific mechanisms and have certain turnover rates. Particulate organic matter (POM), defined as fresh or decomposing organic material (mainly composed of fine root fragments and other organic debris) between 53 and 250 µm in diameter and serves as a readily decomposable substrate for soil microorganisms (Mrabet et al., 2001). Particulate Organic Carbon (POC) responds in a short time period to the management-induced alterations. It can be used as an early indicator of SOM changes since they are difficult to detect, mostly only in long-term experiments (Chen et al., 2009. Thus, it is a useful index of microbially-important SOM because it consists of recognisable organic matter that can be isolated from mineral soils, and it is sensitive to changes in soil management (Franzluebbers, 2000). In addition, particulate organic C constitutes 8 to 25% of Total Organic Carbon (TOC) (Chan, 2007) and represents a transitional stage in the transformation of plant residue to soil C storage (Mao & Zeng, 2010). Therefore, in the long-term, an increase in POC translates into an increase in TOC (Cambardella & Elliott, 1992).

Principle

The method (Cambardella & Elliot, 1992) is based in the soil physical fractionation, the underlying principle is that the association of soil particles and their spatial arrangement play a key role in SOM dynamics, because bioaccessibility is a prerequisite for decomposition. Physical fractionation of SOM is useful for distinguishing specific C pools responsive to management, identifying the physical control of SOM, and characterising the relationship between SOM and the size distribution of aggregates. The difference between total SOC and POC will give us another C fraction: MOC (mineral associated C) which is the SOM chemically stabilised on silt and clay surfaces. However, this is a more stabilised SOM than the POM, and therefore less sensitive to soil management.

Reagents

- Sodium hexametaphosphate (5 g l^{-1}): dissolve 5 g of sodium hexametaphosphate in distilled water, complete to 1 l and shake well

Materials and equipment

- Reciprocal shaker
- 0.053-mm sieve
- Porcelain crucibles 10–15 cm diameter
- Whatman filter paper 541. Hardened Ashless. CAT No. 1541-125
- Oven
- Agate mortar

- Balance
- Distilled water
- Wash water bottle

Procedure

a. Air-dry soil samples

b. Sieved at 2 mm

c. Weight 20 g of dry mineral soil and dispersed by shaking overnight in a 100 ml solution of sodium hexametaphosphate (5 g l^{-1}).

d. Sieved the mixture through a 0.053-mm sieve and gently washed with deionised water (use 1 l of water approximately) the material retained above the sieved

e. Weight the filters

f. Filter the sample.

g. Introduce the filter + soil in the oven and dry at 60º C for 24 hours.

h. Weight the filter+ soil once dry

i. Remove the stones (in case you have) by hand.

j. Weigth the stones (in case you have)

k. Ground the soil using a mortar and stored for carbon analysis. Concentrations of C in the isolated fraction will be determined using a C and N analyser (see section 2.2.7).

Calculations

Soil C in the POC fraction (g C g^{-1} soil) is calculated by multiplying the dry mass of POC (g POC g^{-1} soil) by the respective C concentration (g C g^{-1} POC).

Table 2.2.8.1 Range of values in different agricultural soils

POC (g kg^{-1})	Land use	Soil type	Reference
15–35	Barley/ CT vs NT	Vertisol	Somasundaram et al., (2017)
1.60–4.6	Barley/CC vs NT	Hypercalcic calcisol	Blanco-Moure et al., (2013)
0.58–1.53	Barley/wheat	Calcaric cambisol	Moharana et al., (2017)
2.7	Olive	Hypercalcid calcisol	Martínez-Mena et al., (2008)
0.8	Vegetable cropping system	Gleyc Luvisol	Baiano & Morra (2017)
1.44–4.57	Chestnut orchards	Dystric Cambisols	Borges et al., (2017)

Remarks

- Mineral associated carbon can also be obtained with this method, if the sample passing through the 0.053 mm sieve is collected
- To dry the filters more rapidly, a system can be used to drain the filter before putting it on the oven
- The time in the oven will depend on the quantity of POC to be obtained. 24 hours is the minimum
- As an alternative to removing the stones by hand (step i in the procedure) the dry soil could be sieved with a 1 mm sieve

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35. Soil greenhouse gas emissions

Kristiina Regina[a], Roman Hüppi[b]

[a]Natural Resources Institute Finland (Luke), Jokioinen, Finland

[b]Department of Environmental Science, Institute of Agricultural Sciences, ETH Zurich, Zurich, Switzerland

Importance and applications

The fluxes of nitrous oxide (N_2O), methane (CH_4) and carbon dioxide (CO_2) from soils are indicators of microbial activities in soils. The values measured can be used to estimate the losses of nitrogen and carbon from soils as well as the impact of agricultural production on global warming.

Principle

This method is used to determine gaseous fluxes between the soil and atmosphere. The origin of the flux is microbial metabolism as part of elemental cycles. The principle is to cover an area of soil surface with a measurement chamber and follow the change of gas concentration in the chamber during its enclosure. For this, samples are taken for laboratory analysis or drawn to an analyser in the field to determine the concentration of a gas in the chamber at a defined point in time. The gas flux rate is determined as a function of the concentration in time.

Reagents

- Gases for the gas chromatograph as required by the measurement method
- Reference gases of known concentration

Materials and equipment

- Frames for gas-tight use of chambers
- Vented chambers for gas sample collection. Vent is a 0.5–1 m tube with a diameter of 3–4 mm. Mixing of air during sampling can optionally be achieved by a battery-operated fan or by applying a perforated sampling probe extending from the top to the bottom of the chamber
- Sample vials (glass with rubber septa)
- Syringes with needles
- Stopwatch
- Gas chromatograph or a flow-through instrument for gas analysis

Procedure

N_2O and CH_4 fluxes:

a. Take the chambers to their positions but do not close them yet
b. Close the first chamber and start the stopwatch. Take the first sample from the air inside the chamber
c. Close the second chamber and take the sample 1 or 2 minutes after the first sampling. The time depends on the distance of the chambers and the time available before the next sampling point. Enough time must be allowed to walk between the chambers. Repeat this procedure as long as the first sample of every chamber has been taken

d. Start the second round of sampling. Proceed the same way as during the first round but mix the air in the chamber by filling and emptying the syringe 3-5 times before taking the sample (if there is no fan in the chambers)
e. Proceed with all 4 rounds as above. Options for timing of sampling rounds include e.g. 0, 15, 30, 45 minutes or 0, 20, 40, 60 minutes
f. Make notes of the air temperature and any deviations of the timing etc. Also remember to take the reading of frame height from the soil surface for determining the total headspace volume
g. Take the samples to the laboratory and proceed with the analysis as defined in your laboratory guidelines

CO_2 flux from soil respiration:

a. Install the chamber on place
b. Take the recordings of CO_2 concentration for 1–3 minutes (or as needed for your instrument) and air temperature during the sampling
c. Repeat the procedure for each chamber

Install the chamber frames at a depth of at least 15 cm to keep the roots out of the chamber area and remove all growing plants the day before the measurement. If it is not possible to install the frames before root growth, take into account that the trenched roots may cause overestimation of soil respiration which may require correction in the calculation phase. Respiration from living roots should be excluded but the dead roots from the previous growing season should be present in the measured plot.

Calculations

N_2O and CH_4 fluxes:

A script for case-wise linear or non-linear method selection is used for calculating the results. It implements a selection algorithm using the minimum detectable flux for selecting between the linear and non-linear calculation (Hüppi et al., submitted).

a. Determine the minimum detectable flux of your measurement system at 95% confidence level as instructed in Appendix 3 of De Klein and Harvey (2015) or using the function in the gas fluxes R-package.
b. Calibrate the detector (concentration vs. peak area). Use a reference gas range similar to that of the samples.
c. Using the calibration curve, first calculate the sample concentration as µmol mol^{-1} (ppm). Sample concentration (µmol mol^{-1}) is divided by the volume of ideal gas (l mol^{-1}) yielding the chamber air concentration as µmol L^{-1}. Multiply the concentration (µmol L^{-1}) by the molar weight of the gas (g mol^{-1}) to convert the unit to µg L^{-1}.

   The volume of ideal gas must be temperature-corrected, thus correct for the chamber or air temperature using the ideal gas law. The volume of ideal gas is calculated as V = 0.082056 × (273.15 + T), where 0.082056 is the gas constant (R) and T is the chamber or ambient air temperature (°C).

   R=PV/nT; at standard temperature and pressure R= 1 atm × 22.414 litres/1 mol ×273.15 K = 0.082056 l atm mol^{-1} K^{-1}

d. Use the gas fluxes R-package to calculate the fluxes for each chamber from the sample concentration values. For this you will need the headspace volume, frame area and time of sampling from the start of the chamber enclosure. The script will provide both a linear and “robust” non-linear flux estimate

In the winter, the chamber volume needs to be corrected for the volume of snow inside the chamber. For this, sample a known volume of snow and based on this information divide the snow mass in the chamber with the density of ice (0.9168 g cm^{-3}).

The measurement data is uploaded to the project cloud in the form of final fluxes, unit mg N_2O m^{-2} h^{-1} or mg CH_4 m^{-2} h^{-1}. This method also provides the estimate of ecosystem respiration if your gas chromatograph measures CO_2.

A minimum of three time points are needed for each flux calculation.

CO_2 flux:

The flux rates will be calculated from the concentration data as above for N_2O and CH_4.

The gaps between measurements will be filled with hourly estimates of soil respiration using empirical modelling for each measurement plot based on the temperature-dependence of soil respiration (Lloyd & Taylor, 1994). Hourly soil temperature measurements (depth of 5 cm) are used for this purpose.

$$R = R_{10}e^{E_0\left(\frac{1}{283.15-T_0}-\frac{1}{T-T_0}\right)} = R_{10}e^{308.56\left(\frac{1}{56.02}-\frac{1}{T-227.13}\right)} \quad \text{(Eq. 2.2.10.1)}$$

where

R is the soil respiration;

$\mathbf{R}_{10}$ is the soil respiration at 10°C;

T is the soil temperature.

Further grouping (e.g. seasonal) of the annual measurement data may be needed in order to increase the reliability of modelling. The hourly modelled values are summed to yield daily and annual values.

The measurement data is uploaded to the project cloud in the form of final fluxes, unit mg CO_2 m^{-2} h^{-1}

Remarks

- Every group can use their existing chambers, vials and analysis equipment. Apply one chamber per plot yielding 3–4 replicates. Random chamber placement is recommended but in farmers' fields it is also polite and practical to plan the placement so that e.g. dimensions of spraying equipment are taken into account (so that the farmer can drive between the chamber frames during the growing season).
- As the chambers used for soil respiration measurement are typically smaller than the ones used for N_2O and CH_4 more replicates are needed than for the larger chambers
- For experiments with different subplot areas that are expected to have different emission dynamics (like in vineyards with berms and rows), it is suggested to measure in both conditions on each plot if it is relevant to the experimental treatment
- The enclosure time varies with the chamber dimensions. General advice is found in Klein and Harvey (2015) but the quality of the results tells us if the enclosure was long enough

- The measurements should be taken between 10.00 and 12.00 to avoid bias by temperature. For the same reason, it is also a good practice to vary the chamber sequence between sampling days
- The intended number of samplings per year is 25 and these can be allocated so that periods of high emissions such as fertilisation, flooding, snowmelt, harvest and other abrupt system changes are well represented. It is very important that a measurement is conducted very soon after such an abrupt system change

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36. Pesticides

Violette Geissen, Vera Silva, Hans G.J. Mol, Paul Zomer, Nicolas Beriot, Xioamei Yang, Esperanza Huerta Lwanga, Coen J. Ritsema

Soil Physics and Land Management Group, Wageningen University & Research,

Droevendaalsesteeg 4, 6708PBWageningen, The Netherlands.

Importance and applications

492 active substances for pest and weed control are present in more than 2000 pesticides on the European market with 26 active substances pending (Reg. (EC) No 1107/2009; http://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/public/?event=activesubstance.selection&language=EN). Pesticides enter into the soil as a result of plant protection measures (weed control and pest control). Although the persistence of pesticides has strongly decreased in the last decades, a number of studies describe the occurrence of mixtures of (persistent) pesticides in soils as a result of long-term annual applications (e.g., Organochlorines like DDT and its metabolites, forbidden in 1973 in Europe, Ferencz & Balog, 2010; or Glyphosate and its metabolite AMPA, Gui et al., 2014).

Principle

For obtaining a wide spectrum of pesticides residues in soils, the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method seems to be the most consensual option (Anastassiades et al. 2003, Mol et al. 2008). The extraction with acetonitrile containing 1% acetic acid (ACN 1% HAc) is used to extract organophosphate, organochlorine, carbamate, thiocarbamate, urea, triazine, and other types of pesticides, according to Mol et al. (2008) (Figure 2.2.12.1). For glyphosate and AMPA, column characteristics and instrumentation conditions are followed according to Bento et al. (2016) and Yang et al. (2015) (Figure 2.2.12.1). Therefore the most common pesticides/compounds analyzed with this method are: :Abamectin, Atrazine, Atrazine-deisopropyl, Atrazine-desethyl, Azoxystrobin, Boscalid13C3-caffeine, Carbaryl, Carbofuran, Carbofuran 3-hydroxy, Carbofuran-keto, Chlorpyrifos, Chlorpyrifos-methyl, Cymoxanil, Cyproconazole, Cyprodinil, Diazinon, Difenoconazole, Dimethomorph, Diuron, Epoxiconazole, Ethion, Fenpropimorph, Fluometuron, FluroxypyrImazalil, Imidacloprid, Isoproturon, Linuron, Malathion, Metalaxyl, Metamitron, Myclobutanil, Penconazole, Pinoxaden, Pirimiphos-methyl, Prochloraz, Propiconazole, Prothioconazole, Pyraclostrobin, Quinoxyfen, Simazine, Tebuconazole, Terbuthylazine, Terbuthylazine-desethyl, Triadimenol and Trifloxystrobin.

Reagents

- See Fig. 2.2.12.1

Materials and equipment

- GC-MS, LC-MS, SRM
- Plastic beakers (200 mL)
- Timer

Procedure

Soils should be collected preferably by 2 depths, 0-10, 10-30 cm, and after air dried and sieved < 2 mm, they need to be preserved at -18 ºC until the determination of pesticides is done. For the extraction, soil samples have to be homogenized and then an aliquot of 5 g must be obtained for the determination of the multi-residue method, while two aliquots of 2 g must be taken for the determination of glyphosate and AMPA. Some of the aliquots should be spiked, with the purpose of checking the overall procedure in the liquid chromatography tandem mass spectrometry (LC- MS /MS) analysis (Silva et al. 2019).

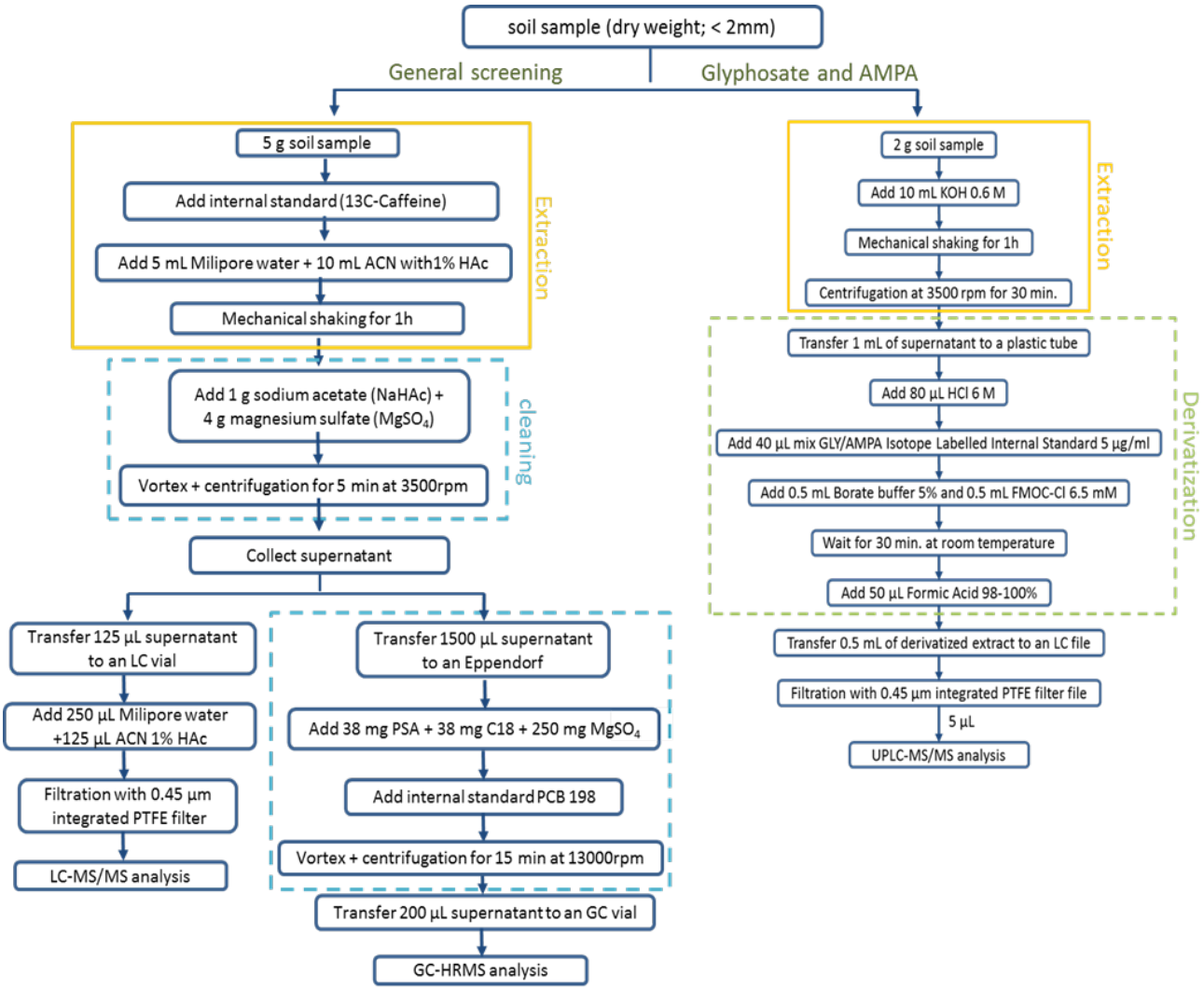

Figure 2.2.12.1 Description of pesticides analysis. General Screening (left) and Glyphosate and AMPA (right). Reagents and procedure

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