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# Comparison of infrared spectroscopy and laser granulometry as alternative methods to estimate soil aggregate stability in Mediterranean *badlands*

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## ABSTRACT

Soil aggregate stability is a key indicator of soil resistance to erosion, but its measurement remains fastidious for large scale uses. Alternative time and cost-effective methods are thus needed. Our objective was to assess and compare the efficiency of laser granulometry (LG) and soil mid- and near-infrared spectroscopy (MIR/NIR) as alternative methods to assess soil aggregate stability in Mediterranean *badland* soils. A collection of 75 *badland* soil samples was used, showing wide variations in soil aggregate stability. Three different categories of measurements were performed: (i) the aggregate breakdown kinetics of the [<1 mm] size fraction under stirring and sonication, tracked by repeated particle size distribution measurements, using LG, (ii) mid-(diffuse-MIR-DR and attenuate transmitted reflectance — MIR-ATR) and near-(NIR-DR) infrared spectra of the fine soil fraction [<2 mm] and (iii) the soil aggregate [3–5 mm] stability, using the standardized method (ISO/FDIS 10930, 2012). Partial least squares regression models were used to predict soil aggregate stability using LG data and infrared spectra. Results showed that NIR-DR and MIR-ATR data provided the best prediction model for soil aggregate stability values (RPD = 2.61 & 2.74; $R^2 = 0.85$ & 0.87), followed by MIR-DR data (RPD = 2.24; $R^2 = 0.89$) and finally LG data (RPD = 2.12; $R^2 = 0.80$). For a quantitative use of the models to assign soil samples to standardized soil aggregate stability classes (ISO/FDIS 10930, 2012), infrared spectra also provided the best accuracy, with a misclassification rate below 30% for NIR-DR and MIR-ATR models, while it reached 43% with the LG-based model. The combination of IR and LG data did not yield a better prediction model for soil aggregate stability values and classes. Infrared-based method also provided best results in terms of time-saving strategy, reducing the measurement time to 8 min only. To conclude, infrared spectra (NIR-DR and MIR-ATR) outperformed LG-data to predict soil aggregate stability. Further development of this technique would require calibrating a set of soil-type specific prediction models for a wide range of soil types.

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## 1. Introduction

Soil aggregation is a key process in soil structuration, influencing a wide range of soil properties, such as water holding capacity, water availability to plant roots and organic carbon sequestration (Bronick and Lal, 2005; Zhao et al., 2007). The stability of soil aggregates, referring to their resistance to breakdown under disruptive forces, is also a good indicator of soil resistance to erosion (Barthès and Roose, 2002). Surface erosion by water is important in Mediterranean regions (García-Ruiz et al., 2013), exhibiting frequent intense rainfall events (Borga et al., 2014) and the occurrence of badland areas. Badlands refer to patches of the landscape characterized by a dense network of ephemeral ravines incising soft erodible rocks with poorly cohesive soils and minimal vegetation (Gallart et al., 2013). In such context, soil aggregate stability offers a possibility to assess soil erodibility without running expensive and time-consuming field runoff experiments (Le Bissonnais, 1996).

Several methods have been developed to measure soil aggregate stability (Chepil, 1952; Kemper and Rosenau, 1986; Le Bissonnais, 1996; Amezketa, 1999). To allow comparison between studies, a

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*Abbreviations:* MWD, mean weight diameter; SOC, soil organic carbon; NIR, near infrared; MIR, mid infrared; DR, diffuse reflectance; ATR, Attenuated total reflectance; PLS, partial least squares; RPD, ratio of performance to deviation; RMSECV, root mean square error of cross-validation; RMSEP, root mean square error prediction; d10, the tenth percentile; d50, the 50th percentile (median); d90, the 90th percentile.

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standardized protocol, composed by three disruptive tests accounting for slaking, mechanical breakdown and physico-chemical dispersion was recently proposed (ISO/FDIS, 10930, 2012), from the work of Le Bissonnais (1996) and Le Bissonnais and Arrouays (1997). However this standardized method remains time-consuming. Labor-intensive steps of this method mainly relate to the numerous manual steps, including wetting and several sieving steps (Rawlins et al., 2013). In soils containing more than 10% of coarse soil particles, such as gravels or marly platelets, an additional step of manual removal of stones further increases the labor investment (Erktan et al., 2016). This actually limits the deployment of this method to a high number of field sites, while there is a growing interest in quantifying the variation of aggregate stability and related soil properties both in time (Abiven et al., 2009; Algayer et al., 2014) and space (Rawlins et al., 2015).

To reduce time investment in soil aggregate stability measurements, two main alternative strategies have been explored: the use of (i) laser granulometry (e.g. Bieganowski and Witkowska, 2010) and (ii) visible, near and mid infrared spectroscopies (e.g. Chang et al., 2001; Madari et al., 2006; Gomez et al., 2013).

Laser granulometry (LG) offers the possibility to run quick, easy and reproducible measurements of soil effective particle size distribution (PSD) of a soil sample in circulating water (Arriaga et al., 2006; Ryzak and Bieganowski, 2011; Sochan et al., 2012). Several alternative methods for time-effective soil aggregate stability measurements were developed using this technique (Beuselinck et al., 1999; Bieganowski and Witkowska, 2010; Schomakers et al., 2011; Grangeon et al., 2012, 2014). The most recent developments of the LG-based technique for rapid and reproducible soil aggregate stability assessment were conducted by Rawlins et al. (2013, 2015). Running continuous PSD measurements, they tracked mean weight diameter (MWD) variations of soil aggregate samples (1–2 mm), progressively disintegrating in circulating water due to hydrodynamic forces and sonication. Soil aggregate stability was assessed by the difference in the continuous size distribution of water stable aggregates and the disaggregated material. The main advantages of the LG-based technique for soil aggregate assessment rely on (i) accounting for a wide range of soil fractions, (ii) tracking soil disintegration by continuous measurements, (iii) limiting the bias related to the operator, and (iv) being less labor-intensive.

Visible, near and mid infrared (Vis, NIR and MIR) spectroscopies have proved to be effective alternative methods to conventional laboratory analyses for assessing several soil properties (Viscarra Rossel et al., 2006), especially biochemical characteristics (O'Rourke and Holden, 2012; Cécillon et al., 2012; Yang et al., 2012). By contrast, studies investigating the prediction of soil structural characteristics are still scant and contradictory (Cécillon et al., 2009, Askari et al., 2015). Nevertheless, recent studies found promising, but still relatively inaccurate results for the prediction of soil aggregate stability using infrared spectroscopy (Madari et al., 2006; Cañasveras et al., 2010, Gomez et al., 2013). Soil aggregate stability is influenced by several soil components, such as soil carbon concentration, calcium carbonates, clays and iron oxydes, which are all well predicted by infrared spectroscopy (Malley et al., 2004; O'Rourke and Holden, 2012; Gomez et al., 2013) and may explain the high potential of this technique to predict soil aggregate stability accurately.

Despite increasing effort in the parallel development of LG and infrared spectroscopy as time and cost-effective methods for determining soil aggregate stability, no study attempted to compare directly these two methods.

The objective of this study was to assess and compare the efficiency of LG and soil infrared spectroscopy (NIR/MIR) as alternative methods to assess soil macro-aggregate stability in Mediterranean *badland* soils. It was hypothesized that (i) NIR and MIR spectra are suitable for prediction of soil macro-aggregate stability due to their ability to detect key drivers of soil aggregate stabilization, such as soil organic carbon (SOC), clays and carbonates, and that (ii) the disintegration of the [<1 mm] soil aggregates under stirring and sonication within the LG is correlated to the disintegration of the [3–5 mm] aggregates after rapid immersion in water, constituting the standardized method of soil macro-aggregate stability measurement.

## 2. Material and methods

### 2.1. Study site and soil sampling

The soils used in this study originate from Mediterranean *badlands* in the Southern French Alps, near Digne-les-Bains at the Draix-Bléone Environmental Research Observatory (http://oredraixbleone.irstea.fr/; 44°08′N, 6°20′E). The site is characterized by a Mountainous and Mediterranean climate (Vallauri, 1997), with an annual mean air temperature of 10.3 °C and an annual mean precipitation of 900 mm (Mathys, 2006). Precipitation regime is heterogeneous, with most rainfall concentrating in May and September during intense storm events (Mathys, 2006). The soils used in this study are Calcaric Leptic Regosols (WRB, 2014) formed above a regolith layer, composed by coarse particles, called platelets (Maquaire et al., 2002) originating from the weathering of Jurassic black marls (superior Bathonian and Callovo-Oxfordian levels), occurring under freeze and thaw cycles (Mathys et al., 2003). The low cohesion of this regolith layer makes it particularly vulnerable to soil surface erosion by water.

Soil samples were collected in the Bouinenc catchment, characterized by a total surface area of 40 km$^2$ and an average altitude of 862 m. In May 2012, in a 3 km$^2$ area within this catchment, 75 independent plots of 1 × 2 m were chosen along a plant succession gradient occurring in gully beds and known to be associated with wide variations in soil aggregate stability (Erktan et al., 2016). Five successional stages were selected according to their dominant vegetation cover. From early to late successional stages, plots were dominated by: (i) herbs (Herbs), (ii) shrubs (Shrub), (iii) small sized trees (STree), (iv) tall sized trees (TTree) and (v) forests (Forest). For each successional stage, fifteen plots were selected and sampled.

In each plot, two composite soil samples were harvested. One sample was collected with a cylindrical core to a 5-cm depth for soil aggregate stability measurements. A second sample was collected for infrared and granulometry analyses as well as basic soil characterization (Erktan et al., 2016). Each composite soil sample was obtained by pooling and homogenizing three sub-samples collected randomly in the plot.

### 2.2. Basic soil characteristics and soil aggregate stability measurements

Basic soil characterization was run on the air-dried soil fraction below 2 mm. The values for several parameters: soil organic carbon (NF ISO 10694, 1995), $CaCO_3$ (NF ISO 10693, 1995) concentrations and soil texture (percentage of clays [0–2 μm], silts [2–50 μm] and fine sand [50 μm–1 mm] measured by LG) were reported in Erktan et al. (2016) and in Table 1. Additional soil parameters for the 75 soil samples, namely total soil nitrogen concentration (Dumas, 1831), and pH in KCl ($pH_{KCl}$; NF ISO 10390, 1994) are reported in Table 1.

Soil aggregate stability was measured on the 75 soil samples, following the standardized method ISO/FDIS 10930 (2012) derived from

**Table 1**
Repartition of the 75 soil samples within the five classes of soil aggregate stability, defined by the norm ISO/FDIS 10930 (2012), from Le Bissonnais (1996).

| Stability class | MWD (mm) | Number of soil samples | | | | | |
|---|---|---|---|---|---|---|---|
| | | Successional stages | | | | | Total |
| | | Herbs | Shrubs | STree | TTree | Forest | |
| Very unstable | <0.4 | 2 | 3 | 3 | 0 | 0 | 8 |
| Unstable | 0.4–0.8 | 6 | 5 | 3 | 0 | 0 | 14 |
| Moderately stable | 0.8–1.3 | 6 | 2 | 2 | 4 | 1 | 15 |
| Stable | 1.3–2.0 | 1 | 1 | 4 | 5 | 2 | 13 |
| Very stable | >2.0 | 0 | 4 | 3 | 6 | 12 | 25 |

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