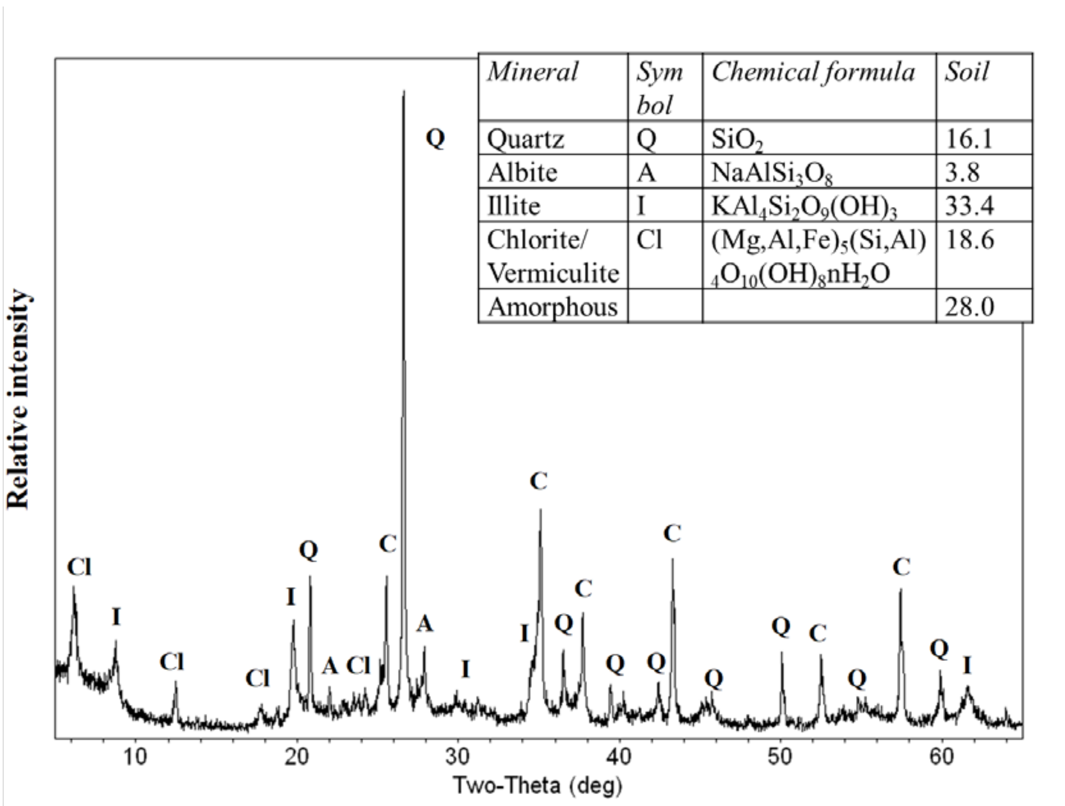
Description of material characterization methods

X-ray Diffraction (XRD)

XRD relies on the interaction of X-rays with crystalline structures, i.e. chemical compounds in which atoms are arranged in space in a predictable manner. Most soil components, such as quartz, feldspars, micas, clays and others, are crystalline structures that can be identified by XRD. Additionally, a wide variety of materials, such as cement, ceramics and various waste types consist of characteristic crystalline compounds that can be identified by XRD. Specifically, X-ray Powder Diffraction (XRPD) is used to analyze materials of mixed composition. The XRPD analysis involves the drying and pulverization of a small amount of solid (typically 10 g) to obtain a representative sample of 1 g. The subsample is then analyzed using a dedicated XRD equipment. The obtained spectrum (Figure 1) contains peaks as a function of the diffraction angle; these are then matched by comparing the observed peak positions with peak positions of pure compounds that are reported in the database of the International Centre for Diffraction Data.



Mineral	*Symbol*	*Chemical formula*	*Soil*
Quartz	Q	SiO_2	16.1
Albite	A	$NaAlSi_3O_8$	3.8
Illite	I	$KAl_4Si_2O_9(OH)_3$	33.4
Chlorite/ Vermiculite	Cl	$(Mg,Al,Fe)_5(Si,Al)_4O_{10}(OH)_8nH_2O$	18.6
Amorphous			28.0

FIGURE 1: EXAMPLE OF XRD PATTERN AND ANALYSIS RESULTS OF A SOIL SAMPLE

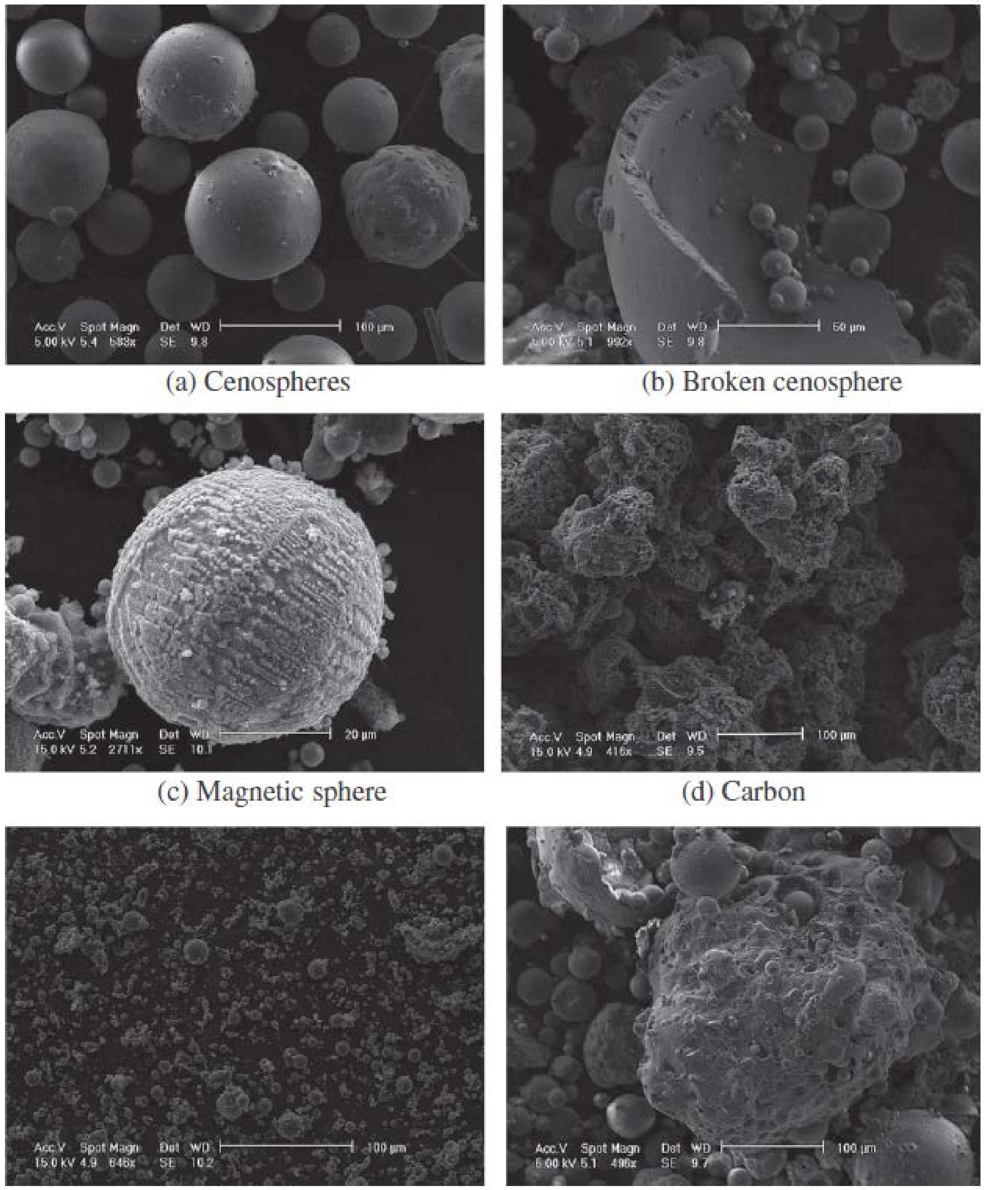
It is also possible to conduct quantitative analysis of the identified compounds, as well as of the total amount of amorphous (non-crystalline) compounds that are present in the solid. An example of a soil analysis with XRD is shown in Figure 1. The obtained composition can be compared with previously reported compositions of various materials in order to identify the likely type and/or source of the analyzed XRD sample. The detection limit for individual minerals is between 1% and 5% wt., depending on the type of sample and the quality of the scan; typically it is in the range 1-2 wt.%.

Scanning Electron Microscopy- Energy Dispersive X-ray (SEM/EDX)

SEM is an imaging method that shows the morphology of materials at the micron level. A typical SEM provides a wide range of magnifications and can visualize materials from the cm to the nm level (magnification of 10X to 500,000X or more). The actual useful range of magnifications that can be achieved depends on the equipment and the material itself.

In terms of geoenvironmental applications, the SEM can be used to obtained images of particle morphology and identify particles of characteristic shape that are linked to a certain type of material. For example, fly ash is known to consist of perfectly spherical particles called cenospheres (see Figure 2), while chromite crystals (characteristic of Chromite Ore Processing Residue) have a pyramidal shape with trigonal faces. Some crystals such as chromite may be identified by both XRD and SEM, while others, like cenospheres are only identified by SEM.

EDX is an accessory of SEM that analyzes the chemical composition of identified particles and can thus help narrow down the nature of the particles (e.g., cenospheres are made up of Si alone, chromite is made up of Mg, Fe and Cr).



(a) Cenospheres (b) Broken cenosphere

(c) Magnetic sphere (d) Carbon

(e) Fine IFA residue (f) Coarse IFA residue

Figure 2: Example of SEM images of fly ash particles (Blissett and Rowson 2012. A review of the multicomponent utilization of coal fly ash, Fuel, 97, 1-23)