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Quantification of complex mixtures using the PONKCS-assisted Rietveld refinement method

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Quantitative mineralogy of mixtures – containing clay minerals or other complex phases – still encounters a lot of problems. Contrary to other geological analysis methods (*e.g.* to determine the chemical composition), the accuracy of XRD based quantitative mineralogy is significantly less. Most of the problems causing the misfit are related to the crystal structure. In clay minerals, crystals often exhibit disorder or substitution of ions and in most cases, even the exact crystal structure is unknown.

Today most researchers use either the Rietveld or the full pattern fitting method for the analysis of XRD patterns. However, both methods have shortcomings when dealing with complex minerals like clays and amorphous phases. Ten years ago, the Partial Or No Known Crystal Structure (PONKCS) method was reintroduced by Scarlett & Madsen (2006). This method is a combination of the Rietveld method (crystal structure based) and the full pattern fitting method (reference sample based), and uses a crystal structure based reference pattern in the Rietveld space. The advantage of the PONKCS method is that it allows to generate a structure file for phases where no, or only partial, crystal structure information is available.

In this study, the suitability of the PONKCS method to carry out an accurate quantification of mineral mixtures with complex phases was investigated by using a set of reference standards, both consisting of clay minerals and amorphous phases. Three issues were 'tackled' prior to the effective analysis. First, to avoid preparation related bias, the sample preparation method – as described by Środoń et al. (2001) – was complemented with a spray drying treatment. This additional step significantly decreased the tendency for preferred orientation of platy minerals. Second, samples spiked with different amounts of the internal standard zincite, were used to calibrate the PONKCS patterns for quantification. Third, since the PONKCS method assumes that the background is solely device dependent, a limited dataset of simple oxides was measured to effectively test their influence on the background. The XRD patterns indeed show higher background levels for oxides with lower mass absorption coefficients.

Testing the accuracy of a quantitative mineralogy technique is in most cases a challenge since the real composition is rarely known. Artificial mixtures were therefore prepared to compare the real content with the content calculated from the quantitative analysis. In the four mixtures examined, the individual complex minerals could be accurately quantified with an average absolute error of 1.0–2.0 wt% misfit for crystalline components, 1.6 wt% for the clay minerals and 2.0 wt% for the amorphous phase. These results are significantly better than those obtained by the 'top 10' participants of the *2016 Reynolds Cup Competition* (international mineralogy round robin). Their average absolute error was 2.2 wt% misfit for crystalline components, 3.0 wt% for the clay minerals and 3.2 wt% for the amorphous phase.

From this study, it can thus be concluded that the PONKCS method has great potential and is very promising for the accurate quantification of complex mineral mixtures. It has clearly more advantages and less disadvantages compared to other techniques presently used for quantitative mineralogical analysis.

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Paleostress analysis of Cretaceous rocks at Flamborough (Yorkshire, UK)

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In the chalky area of the North of England (Northern Chalk Province) bordering the North Sea, Cretaceous formations of the promontory of Flamborough have been the object of numerous structural studies (Peacock & Sanderson, 1993; Sagi et al., 2016). Nevertheless, these fractured rocks have never been analysed in terms of paleostress tensors despite the multiplicity of existing tectonic markers. So, a structural survey along the 17 km of cliff was carried out. The aim of this study was to assess the normal, reverse and strike-slip fault population, discriminating their neoformed or inherited character with a view to evaluate the paleostress tensors (Angelier, 1990). Moreover, other tectonic markers have been also located and measured such as slickolithe faults, tension gashes, breccia faults and major tectonic joints (master joints). All of these structural objects have been systematically listed in every affected formation in order to propose a geodynamic evolution.

Accordingly, the microtectonic history recorded in the Yorkshire chalk is progressive and multi-stage due to the presence of two major regional structures (Glennie & Boegner, 1981; Kirby & Swallow, 1987). First, the Dowsing Fault zone (NW-SE) which limits the offshore Sole Pit Basin, and is located in the east of the promontory. The second structural weakness is an E-W axis, the Howardian-Flamborough Fault Belt outcropping at Selwicks Bay. Apart from these major faults, the geodynamic environment of the North Sea was very active during the Mesozoic and Cenozoic Eras (Glennie & Boegner, 1981; Kirby & Swallow, 1987). Indeed, during the late Cretaceous and Cenozoic there is a kinematic shift across the European Northwest, from a globally extensive regime, into a compressive regime with two inversion episodes (Laramide and Alpine inversions) when a lot of sedimentary basins were uplifted and eroded.

Three compressive directions are remarkably well recorded at Flamborough (N-S, NW-SE, NE-SW) mainly represented by strike-slip faults. Particularly, the Cenozoic N-S compression, but this analysis also highlighted a NW-SE compression (Eo-Alpine) pre-dating the Cenozoic inversion due to the reactivation of the regional major lineaments.

At the same times as these compressive regimes, three extensive directions (E-W, NE-SW and NW-SE) are observed. They are correlated relatively well with the opening dynamics of the North Sea Basin and European Northwest. As highlighted by others authors, the promontory faulting to the north and to the south is different and without any doubt this faulting is linked to the presence of regional faults. Nevertheless, it should be noted that these chalky formations exhibit different geomechanical behaviours (Welch et al., 2015), undoubtedly linked to tectonic stresses in connection with the presence of regional faults.

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Evolution of the Andaman Basin: constraints from thermochronometry of the Andaman-Nicobar and Peninsular Thai margins

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Together with the Andaman-Nicobar Islands, the Andaman basin forms a part of a major arc-trench system. The Andaman basin is located in Southeast Asia, between the Sunda subduction trench and Peninsular Thailand and Malaysia and stretches from Myanmar in the north to Sumatra in the south. According to Khan & Chakraborty (2005), a two-phase mechanism opened the Andaman basin in which, during the final phase in the Miocene-Pliocene, subduction slab steepening, combined with the strike slip movement along the Sagaing fault zone, caused enough extensional force for spreading to occur.

Thailand consists of four major continental blocks (Indochina, Sukhothai arc, Inthanon zone and Sibumasu) that were once part of Gondwana and accreted during the Indosinian orogeny. Thailand is crosscut by several major fault systems such as the Three Pagodas and the Mae Ping fault zones and their extensions Ranong and Khlong Marui fault zones, respectively. Lithologically and geochronologically, three major granite belts can be distinguished in Thailand, designated as the Eastern, Central and Western granitoid belts.

Two areas were sampled: one area is located on Peninsular Thailand and one area on the Andaman Islands. For Thailand, 10 granite samples of the Western granitoid belt and one of the Central belt were analysed by means of apatite fission track thermochronology (AFT) using two different protocols: the external detector method (EDM) and the Laser ablation inductive coupled plasma MS (LA-ICP-MS). Both methods were then compared to each other. Six samples of the most differentiated gabbroids of the Andaman ophiolites were analysed by means of Zircon U-Pb dating (ZUPb) and AFT using the LA-ICP-MS.

AFT-ages obtained for the Thailand samples using the EDM are ranging between from 33 Ma to 11 Ma which can be divided in two intervals: 33–25 Ma (Oligocene) and 20–11 Ma (Miocene). The ages obtained with the LA-ICP-MS give the same range and intervals. However, two samples, located at the Ranong Fault zone, showing Miocene ages with EDM, show Oligocene ages with the LA-ICP-MS. The younger ages are located in the region between the Khlong Marui and the Ranong Fault zone and can be attributed to the opening of the Andaman basin. The older ages are located at the SE side of the Khlong Marui fault or at the NW side of the Ranong fault and coincide with offshore basin formation caused by the reactivation of the Khlong Marui and Ranong fault zones (Watkinson et al., 2011). This theory is advocated by the results obtained with the LA-ICP-MS. The results provided by the EDM on the other hand could indicate a possible local event or a continuation of the last deformation event described by Watkinson et al. (2011). Thermal history models obtained for these samples show three stages. The first stage (until 30–25 Ma as given by the Oligocene ages) is a rapid cooling that can be attributed to exhumation caused by the reactivation. During the second stage, no thermal activity occurred except for a possible minor reheating that could be caused by extension and hence an increase in thermal gradient. The last stage (starting at 10–5 Ma as shown by the Miocene ages) is a cooling to present day surface temperature that can be related to the opening of the Andaman basin which causes a base level drop and erosional denudation of the surrounding margins.

The ZUPb ages obtained for the Andaman gabbroids show late Cretaceous ages (93–73 Ma) and are considered to be the crystallisation age of these gabbriods. According to Pal (2011), the geochemistry of the Andaman ophiolites indicates suprasubduction zone spreading. These spreading axes formed shortly after the initiation of subduction and therefore advocate the proposed start of subduction by Chatterjee et al. (2013). According to the obtained ZUPb-ages, volcanic activity should have continued until at least the late late Cretaceous which underscores Pal (2011) statement where igneous activity ceased around the Cretaceous-Paleocene boundary.

Due to low quality and quantity of the apatites in the Andaman gabbroids only two samples gave AFT ages. One sample shows Miocene age of 14 Ma and the other sample shows an age of 42 Ma. The younger age can be ascribed to the opening of the Andaman basin, as for the Thailand samples. The sample with the older age showed low U-content (an average of <4 ppm) and therefore need to be interpreted with caution. However, a similar AFT age is found by Allen et al. (2008) and coincides with the emplacement of the ophiolite slices (Pal et al., 2003).

Further research can be done on the correlation between the EDM and the LA-ICP-MS, to draw more rigid conclusion. Also, more research could be performed to determine the cause of the age deviation between both techniques and hence diminish its influence. Since the Andaman Island did not provide a lot of data, more samples should be analysed to obtain a better impression of the thermal evolution. Iridium irradiation can be considered to increase the amount of confined tracks in the apatite minerals which are needed to create thermal history models for the Andaman islands. In general, other geochronological techniques can be performed on these samples to reveal other tectonic events (if there are any) or obtain a more detailed thermal history of the basin margins.

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Geophysical investigations and numerical back analysis of a landslide in the *Poudingue de Malmedy*, located in the Hockai Fault Zone, Belgium

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The ancient Bévercé landslide (region of Malmedy, Belgium) located in the *Poudingue de Malmedy*, a local Permian conglomerate formation including predominantly limestone clasts in an argillaceous-calcareous cementation, was recently discovered through field surveys and the analysis of new LiDAR data collected by the Walloon Region, Belgium. A pronounced (assumed) fault scarp with an orientation of N330°E can be found directly in the North of the landslide, parallel to the Eastern border faults of the Hockai Fault Zone (HFZ). The HFZ is a seismically active zone along which the most important seismic event of NW Europe occurred: the 1692 Verviers Earthquake of magnitude Ms = 6 – 6.3. The fault zone is further recognised by various geomorphic and tectonic markers, such as fault scarps and graben structures, with the Bévercé landslide situated in the SE part.

This work is focused on the thorough landslide investigation by integrating a maximum of methods to comprehend its formation. On the basis of the high resolution LiDAR data, a spatial analysis combined with geomorphological mapping of the landslide area were carried out. In addition, a small geotechnical survey allowed us to estimate characteristic geomechanical parameters of the conglomerate rock. The subsurface of the study area was explored with three distinct geophysical methods: microseismic ambient noise measurements (H/V), seismic refraction surveys (SRT, interpreted in form of P-wave tomographies and surface wave analyses), as well as electrical resistivity tomography (ERT). An integrated 3D geomodel visualises the study site in terms of its surface and subsurface structures on the basis of the collected data. The conglomerate formation can be subdivided into a fractured and an intact unit, the contact between both units being the possible shearing surface on top of which the landslide developed. A 2D section extracted from the geomodel was implemented for 2D numeric modelling to back analyse dynamic slope formation of the Bévercé landslide in the past, i.e. to solve the question if a seismic input could have led to the prominent mass movement. The modelled block displacements underestimate the actual mass movements and are compared to the corresponding Newmark displacement. A short spectral analysis compares modelled to measured site response.

Reservoir characterisation and facies classification of the detrital deposits adjacent to a travertine system in Denizli, Turkey

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The Ballık area is famous for its world-class travertine deposits, located near the city of Denizli (Turkey). The domal travertine deposits are considered to be a possible reservoir analogue for some of the continental carbonates of the Pre-Salt carbonates in the South Atlantic. Lacustrine and detrital sediments are also part of the Pre-Salt play, highlighting the importance of unravelling their mutual relationship. This study aims to describe the different detrital lithologies that are observed adjacent to the continental carbonates and understand their depositional processes.

In total five different facies are observed. Numerous XRD-analyses (both bulk and oriented clay slides) were performed on the lacustrine facies (1), which is composed of rhythmically laminated marls. These analyses lead to the observation of both palygorskite and (non-detrital) dolomite, potentially associated with the evaporitic setting and the pedogenic processes that characterise these sediments. Zoned calcite rims are observed as well, which indicate a systematically fluctuating geochemistry of the fluids present in these deposits. The latter leads to the formation of both bright and non-luminescent calcite rims, which tend to have a preferential orientation. The polygenetic conglomerates of the fluvial facies (2) are characterised by a variable degree of cementation, which is related to the proximity of the travertine system. The latter results in permeabilities ranging from 0.02 to 3000 mD. The debris-flow facies (3) consists of monogenetic travertine breccias and therefore differs from the fluvial conglomerates. This facies shows evidence for gravity-driven processes (e.g. poorly-sorted breccias, angular clasts, mud-supported matrix, coarsening upwards trend, etc.) in the deposition of these slope deposits. The gastropod facies (4) is characterised by isolated moldic pores, resulting in high porosities (8.5 – 14.0%) and low permeabilities (0.05 – 0.25 mD). Finally the coquina facies (5) is characterised by the bioclastic accumulation of shell fragments, caused either by fluvial or shoreline accumulation processes, leading to high porosity and permeability values (40% and 90 mD).

This work has allowed us to characterise five detrital facies in terms of geochemical, mineralogical, reservoir and sedimentological properties. Each of these facies are characterised by a unique depositional setting and several sedimentological parameters (e.g. gravity-driven processes, lake stratification leading to laminated marls, potentially pedogenic dolomite). In the Ballık area, the detrital deposits make up a significant volume of the deposited sediments, highlighting their importance in a reservoir analogue study. This work has further highlighted the depositional processes of the detrital sediments near the continental carbonates in the Ballık area and the implications they have on reservoir architecture.

Reconstruction of the seismic history of Aysén fjord (South Chile) by means of seismic stratigraphy and sediment-core analysis

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Chile is one of the most seismically active regions in the world, with regularly occurring earthquakes of moment magnitudes exceeding 6. These are large earthquakes, capable of generating intense levels of ground shaking over very large distances. They cause a lot of damage to infrastructure and more importantly also loss of human life. In this way they are one of the greatest natural hazards in Chile. Earthquakes in the southern part of the country originate from either the subduction zone running parallel to the Chilean coastline or from the Liquiñe-Ofqui Fault Zone (LOFZ), which is a dextral strike-slip fault running through the northern Chilean fjordland. In 2007, the Aysén region was hit by a seismic swarm originating from this structural lineament. The highest-magnitude earthquake epicentre was located in Aysén fjord, causing seismic shaking that led to tens of landslides. In turn, these generated large displacement waves or tsunamis in the fjord. In spite of the damage, this type of events are highly interesting from a geological point of view, because landslides and other types of mass-transport deposits (MTDs) that enter into a basin are bound to make an imprint in the sediments present on the bottom of the basin. Thousands of years later, these imprints are preserved in the sedimentary infill of the basin as deformed basin-plain deposits and/or associated (mega)turbidites. These deposits form an excellent record of past earthquakes.

The ideal way to find these types of structures is combining seismic data with one or more long sediment cores. In this way, mass movements have proven to be useful for reconstructing earthquake history in several different locations throughout the world. The potential of Aysén fjord for paleoseismological reconstructions of the region had already been revealed by a prior study, but lacked detail. In this research, a dense network of high-resolution airgun, sparker and TOPAS seismic profiles was used to map prehistorical MTDs and determine whether they were seismically triggered. Additionally, a 21 metre long sediment core was analysed for the presence of turbidites and tephras in order to correlate it with the seismic data. Macroscopic and organic matter was retrieved for radiocarbon dating and provided an age model for the seismic events. Modelling of ground motion intensities was applied to provide preliminary constraints on the epicentral location of these events.

This research has once again proven that fjords are an ideal setting to perform paleoseismological studies. Seismic profiling and a sediment core from Aysén fjord have revealed eleven stratigraphic levels at which MTDs are present. The MTDs are of very different types and morphologies and, based on the synchronicity criterion, seven of the identified levels are interpreted as the result of major earthquakes with a high certainty. Six of them are likely linked to the LOFZ with epicentres quite close to the fjord. Another was interpreted as the result of a megathrust earthquake. The remaining four levels with MTDs were probably also seismically triggered, but this could not be verified since they were not retrieved by the sediment core. This also shows why it is important to combine both data types. Correlation of the seismic data with the sediment core showed at least three pre-Holocene events, although an exact age could not be provided due to a limited core length. A rough estimate of one earthquake every 2000 years occurring on the LOFZ in the Aysén area during the Holocene could be established, although large variations can occur. This value is rather low, considering the amount of stress built up in the area due to subduction. This can be attributed to the limited region in which earthquakes can produce sufficient ground motions to be able to leave a trace in the sedimentary infill of the fjord. Traces of earthquakes with an epicentre located at more than about 100 km distance from Aysén fjord will never be present in its geological record. Nevertheless, a recurrence rate for earthquakes related to the LOFZ occurring in the Aysén region has been successfully established.

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