Ber. Inst. Erdwiss. KFUniv. Graz	ISSN 1608-8166	Band 16	Graz 2011	
IGCP 596 Opening Meeting	Graz, 19-24 th September 2011			

The influence of different acid dissolution methods on insoluble residues of limestones and their magnetic properties and mineralogical composition

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Three different acids and dissolution methods were applied to obtaining of acid-insoluble residues of limestones in the Prague Synform, central Bohemia (Lochkov Formation, Lochkovian, Pozar 3 section near Praha-Reporyje). We have studied mineralogical compositions, structures and physical characteristics of these residues, with particular emphasis on the relationships between compositions and applied methods. The techniques of limestone dissolution are routinely used to extract conodont elements, heavy minerals or any non-noncarbonate impurities in limestone at all. The material obtained is then often used for instrumental analyses, e.g. X-ray, mass spectrometric or image analysis methods, and the results might be considerably affected by different techniques of extraction. This study is based on three sets of dissolution experiments using the acetic, formic and hydrochloric acid. Mineralogical and magneto-mineralogical properties of these insoluble residues are related to the whole-rock magnetism and identification of carriers of magnetic susceptibility (MS) can help and better understand the complexity of rock magnetism but also the specific effects of each kind of acid on the residual mineral structures. Also the solution regimes have to be considered.

Light and heavy fractions were studied separately using measurements of magnetic susceptibility (MS), temperature, field and frequency dependence of MS, isothermal remanent magnetization (IRM), saturated isothermal remanent magnetization (SIRM), anhysteretic remanent magnetization (ARM), together with X-ray diffraction (XRD) identification of clay minerals and scanning electron microprobe (SEM) and energy dispersive spectroscopic (EDX) analyses. Moreover the results acquired were compared with whole-rock samples and their parameters to find out the role of dissolution of some important magnetic phases and carriers (e.g. iron oxides and oxyhydroxides) in the acids.

This experimental study extends the results of the previous work on residues from the Lochkovian up to Emsian strata from the same section (KOPTÍKOVÁ et al. 2010) and also the results on the two sets of residues after dissolution in acetic and hydrochloric acids, which were obtained from the S-D boundary beds in the Prague Synform by VACEK et al. (2010).

Hydrochloric acid solution method causes almost complete dissolution of iron oxides (magnetite, hematite grains) but SIRM curve reveals the possibility of their later neoformation. XRD analyses revealed very similar diffractograms but slight differences have been identified, despite the fact that the measured amounts of mineral phases are also influenced by detection limits of these methods (1 to 5%), influences of coexisting phases, degree of crystallinity or preferential arrangement. This was eliminated using SEM-EDX analyses of polished samples of aggregated residues and individual mineral grains. Quartz, clay minerals (illite, kaolinite), feldspars (microcline), micas (muscovite, biotite), pyrite and gypsum were identified as the prevailed phases. Marcasite, pyroxene/amphibole grains or rutile grains occur more scarcely, but still in amounts which must be considered for the rock characteristics. The residues obtained using acetic acid have higher variability in mineral assemblages (clay minerals, micas) if compared to those dissolved in formic or hydrochloric acid. All of the studied samples show an evident dominance of mineral phases with paramagnetic behaviour, and ironbearing minerals of paramagnetic behaviour control the MS signal rather than those having ferromagnetic characteristics (the latter have surprisingly slight contribution to MS, being detected only e.g. by means of IRM).

This study contributes to IGCP 596, IGCP 580, and projects P210/10/2351 and IAAX00130702.

Ber. Inst. Erdwiss. KFUniv. Graz	ISSN 1608-8166	Band 16	Graz 2011	
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