COMPARATIVE GRAIN-SIZE MEASUREMENTS FOR VALIDATING SAMPLING AND PRETREATMENT TECHNIQUES IN TERMS OF SOLIFLUCTION LANDFORMS, SOUTHERN CARPATHIANS, ROMANIA

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Abstract

Grain-size distribution has become in the last years an important indicator in the analysis of periglacial processes and landforms. However, as they exhibit a complex sedimentology, careful sampling is required to draw meaningful conclusions. The aim of the present study was therefore to validate the sampling procedure carried out on solifluction forms and to evaluate the effect of sampling pretreatment during grain size analysis. A comparison between multiple measurements of grain size distribution using the laser diffraction method (LDM) was performed on 54 sediment samples collected from different solifluction landforms at different depths in the alpine area of the Southern Carpathians. The results of parallel measurements were compared using textural and statistical indicators. The received distributions reinforced the properness of field sampling procedure in most of the cases. The results of textural classification and fractional composition showed a high consistency between the two parallel measurements made on untreated and pretreated samples. An overall fining as a matter of etching was identified. Relative deviation increased and correlation decreased as pretreatment advanced. HCl etching resulted a greater deviation and variability in case of the sand fraction, H_2O_2 rather affected the silt fraction. The greatest deviations were experienced in case of landforms developed on crystalline limestone. Pretreatment of samples introduced a major uncertainty to further comparison and interpretation. Thus, multiple LD measurements on a representative group of samples from the entire sample set were suggested before the geomorphological or environmental interpretation of results to decrease the uncertainties and to validate the processes.

Keywords: laser diffraction method, grain size distribution, acid pretreatment, solifluction landforms, Southern Carpathians

INTRODUCTION

Grain size distribution is one of the most important sedimentological parameters (Ryżak and Bieganowski, 2011), representing the percentage of the total dry weight of sediment grains of a given size fraction. Grain size distribution influences other properties such as pore distribution, water retention, water conductivity, soil nitrification, thermal and absorption properties etc. (Ryżak and Bieganowski, 2011), which in turn highly influence alpine solifluctional processes and landforms.

In the last years several new methods were developed for grain-size analysis, including electroresistance counting, photometrical techniques, X-ray attenuation, optical determination using image analysis, time of transition and laser diffraction (McCave and Syvitski, 1991; Beuselinck et al., 1998; Goossens, 2008; Di Stefano et al., 2010). All these new methods generally have the advantage of covering a wide range of grain sizes, using less quantity of sediments, speed in analysis, reproducibility and fewer possibilities for operator failure (Di Stefano et al., 2010; Kun et al., 2013). Among these the use of the laser diffraction method (LDM) seems to be the most widespread, as it is cost effective, its precision and reproducibility are high. LDM is basically based on the dispersion and diffraction of a laser beam on the measured particles. The scattered laser light is recorded on sensors and the diffraction angle in which the beam is scattered is inversely proportional to particle size. The software of the equipment recalculates the information from the sensors into volumetric grain size distribution (Ryżak and Bieganowski, 2011).

The accuracy of the measurement is influenced by many factors, e.g. the color of the suspension, the mineral composition and opacity of particles, or by organic and carbonate content (Kun et al., 2013). Considering that grain size measurements are affected by the applied pretreatment method, there has been a debate on what procedures should be applied. Some researchers still underline the necessity of using acids when the organic content is high (Murray, 2002) while others found this unnecessary (Beuselinck et al., 1998) and stating that ultrasonic dispersion can replace chemical pretreatment and dispersion methods (Ryżak and Bieganowski, 2011).

In earth sciences LDM has mainly been applied on soil samples, loess, lacustrine, marine and, fluvial sediments (Loizeau et al., 1994; Konert and Vandenberghe, 1997; Buurman et al., 2001; Arnaud, 2005; Di Stefano et al., 2010; Ryżak and Bieganowski, 2011; Forde et al., 2012; Kun et al., 2013), and just in the last years starte to be applied on solifluction landforms (Ridefelt and Boelhouwers, 2006; Oliva et al., 2009; Ridefelt et al., 2011).

Grain-size analysis carried out on solifluction landforms so far has beene made on untreated samples, without evaluating the necessity of pretreatment or the sampling strategy.

Applications in alpine environments require more attention regarding that the material from solifluction lobes is disordered and overlapped by slow mass soil moving (Harris et al., 2008). In these circumstances representative and reproducible field sampling can be an important issue and must be validated before drawing further sedimentological or geomorphological conclusions.

The aim of this study thereby was to attest the correctness of sampling in case of Southern Carpathian solifluction landforms using multiple laser diffraction measurements and to evaluate the effect of sample pretreatment on the results.

STUDY AREA AND METHODS

Solifluction sediment samples were collected from the alpine area of Southern Carpathians, Romania, from different mountain ranges (Fig. 1). The Southern Carpathians are the highest sector of the Romanian Carpathians (Moldoveanu Peak – 2544 m a.s.l.) with seasonal freezing conditions in more than 6 months annually (Urdea, 1993). In the alpine area the climatic conditions are rather cold, with negative mean annual air temperature above 2000 m a.s.l. (-0.5°C at Țarcu - 2180 m a.s.l and -2.4°C at Omu -2505 m a.s.l.) and precipitation over 1000 mm. Above the tree line (1700-1800 m a.s.l.) extensive areas are affected by solifluction, whereas other periglacial landforms (block streams, rock glaciers, talus cones and scree slopes, block fields, patterned ground, ploughing blocks, earth hummocks, etc.) are also common.

The Southern Carpathians are in general composed of crystalline schists with granite intrusions, especially in Cindrel and Făgăraș Moutains. Whereas the Tarcu Moutains is primarily built up of granitoides (northern part), limestones (central part) crystalline schists, sandstones and conglomerates. In terms of lithology, Cindrel and Sureanu Moutains belong to the Getic Fabrics (paragneiss, micaschists and amphibolites), while Parâng Moutains are part of the Danubian Unit (granitoides, amphibolites, and limestones). Characteristic soil type in the area is alpine meadow umbrisol, from the typical to cambic, lithic and skeletal subtypes.

A wide variety of solifluction landforms occur in the alpine environments based on their genesis. Most common and widespread are turf-banked solifluction lobes, while so called ploughing blocks are less frequent (Fig. 2). The term solifluction include all the processes (gelifluction, frost creep, frost heaving and frost sorting, periglacial elevation) contributing to slow mass soil movement in a periglacial environment and leading to the formation of solifluction lobes and terraces (Harris, 2007).

Solifluction lobes have a frontal height ranging from several cm to more than 1 m and length from several cm to more than 10 m (Hugenholtz and Lewkowicz, 2002; Matsuoka et al., 2005).
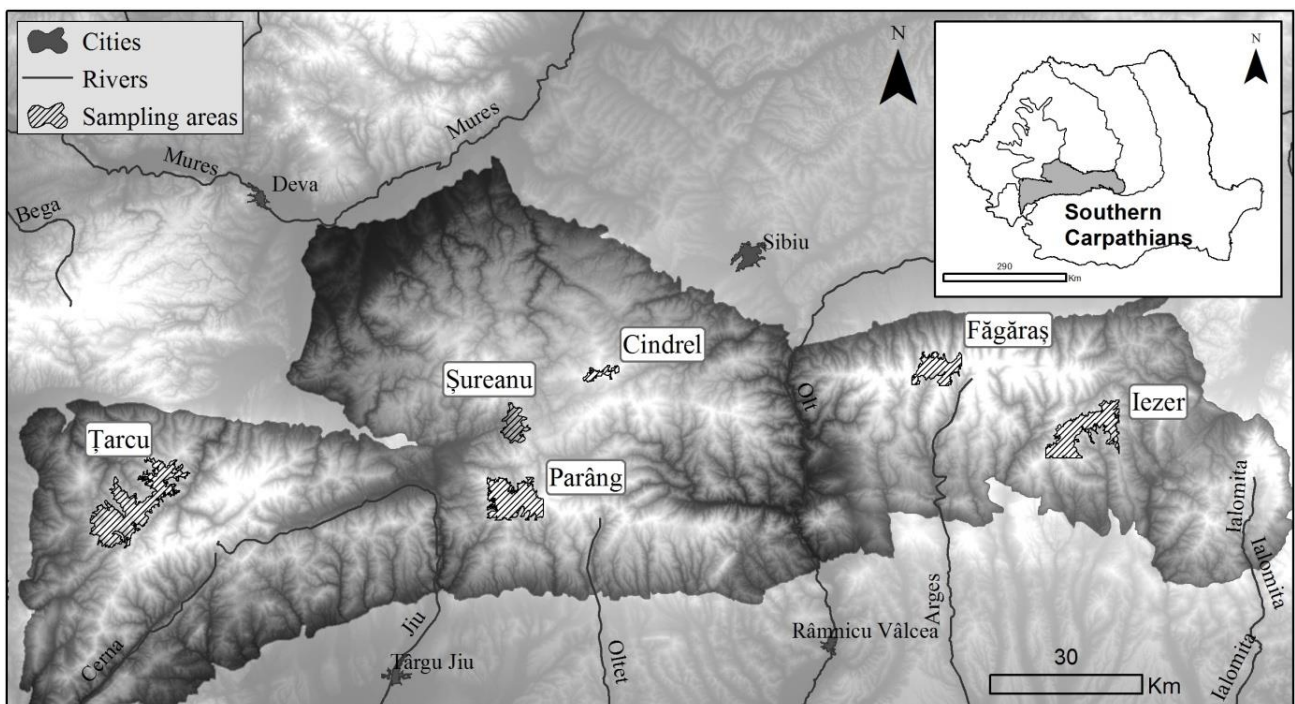

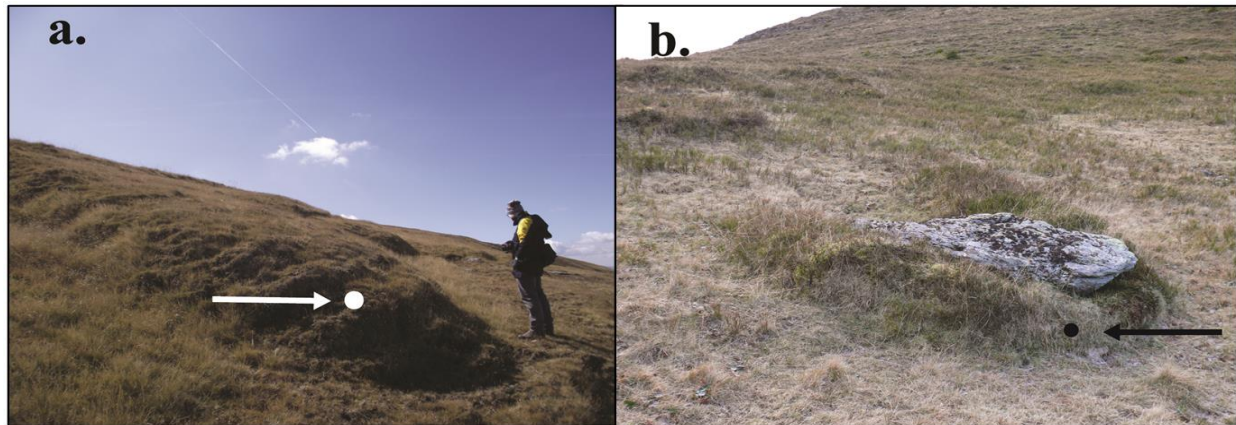*Fig. 1* The location of sampling areas

Fig. 2 Sampling of solifluction landforms: a. turf-banked lobe, b. ploughing block

Ploughing blocks represent a form of mass movement when a block moves downslope faster than the surrounding material, resulting a mound on the front and lateral sides of the block, and a depression behind (French, 1996). Occurence of ploughing blocks is associated with areas of active solifluction and frost-susceptible soils with low plasticity and liquidity limits (Ballantyne, 2001). Their size varies from several cm to almost 5 m (Hall et al., 2001) and alongside the solifluction lobes they represent an indicator of current periglacial phenomena (Ballantyne, 2001; Berthling et al., 2001).

Sampling sites were selected based on their elevation, aspect and geographic location. In all 54 sediment

Table 1 Field and laboratory coding and origin of samples (a - ploughing block, b - turf-banked lobe)

Field ID	Depth (cm)	Lab. ID	Type	Mountain Range	Field ID	Depth (cm)	Lab. ID	Type	Mountain Range
ST136_a25	25	1.	a	Tarcu	C4_25	25	28.	b	Fagaras
T36_a20	20	2.	a	Tarcu	V1_25	25	29.	b	Fagaras
T36_b20	20	3.	a	Tarcu	C2_25	25	30.	b	Fagaras
T36_c20	25	4.	a	Tarcu	P1_25	25	31.	b	Fagaras
T36_d25	25	5.	a	Tarcu	C18_25	25	32.	b	Fagaras
T42_a25	33	6.	a	Tarcu	Pa_D20	20	33.	b	Fagaras
T22_a33	23	7.	a	Tarcu	P8Da_20	20	34.	b	Fagaras
MMlob_23	25	8.	b	Tarcu	P8Da_80	80	35.	b	Fagaras
LC8_25	25	9.	b	Cindrel	P8D_riser2	20	36.	b	Fagaras
LC8_45	45	10.	b	Cindrel	P8Da_60	60	37.	b	Fagaras
LC1_20	20	11.	b	Cindrel	P8Db_25	25	38.	b	Fagaras
I_25	25	12.	b	Iezer	P8Da_40	40	39.	b	Fagaras
I3_35	35	13.	b	Iezer	P8D_riser1	20	40.	b	Fagaras
BRLA12_A28	28	14.	a	Fagaras	Pa19Da_40	40	41.	b	Fagaras
BRLA12_B28	28	15.	a	Fagaras	Pa19Da_60	60	42.	b	Fagaras
BRLA12_C40	40	16.	a	Fagaras	Pa19Da_80	80	43.	b	Fagaras
BRLA12_D15	15	17.	a	Fagaras	Pa19Db_25	25	44.	b	Fagaras
BRLA12_E18	18	18.	a	Fagaras	Pa19Db_50	50	45.	b	Fagaras
S1_25	25	19.	b	Sureanu	Pa19Db2_25	25	46.	b	Fagaras
S1_45	45	20.	b	Sureanu	Pa19Da2_40	40	47.	b	Fagaras
P8_25	25	21.	b	Fagaras	Pa19Da2_80	80	48.	b	Fagaras
Pa18A_25	25	22.	b	Fagaras	Pa19Da_110	110	49.	b	Fagaras
Pa18B_25	25	23.	b	Fagaras	Pa19Da_100	100	50.	b	Fagaras
Pa19Db_riser	20	24.	b	Fagaras	P8Da2_40	40	51.	b	Fagaras
Pa19Da_20	20	25.	b	Fagaras	P8Da_75	75	52.	b	Fagaras
Pa19Db_45	45	26.	b	Fagaras	P8Da2_80	80	53.	b	Fagaras
Pa19Da_105	105	27.	b	Fagaras	LP_25	25	54.	b	Parang

samples were extracted from 17 turf-banked solifluction lobes and from the front mound of 5 ploughing blocks for grain size and other sedimentological analyses (Fig. 2). Sampling depth ranged from 20 to 110 cm for turf-banked lobes and 15 to 40 cm for ploughing block mounds (Table 1.). Samples of approx. 0.5 kg were extracted by digging, thus samples were considered representative for later geomorphological comparisons, but might not be representative for stratigraphic analysis within the form.

All the laboratory work was performed in the sedimentology laboratory of the Department of Physical Geography and Geoinformatics, University of Szeged, Hungary. From each sampling bag two subsamples (Set A and Set B) were extracted from different positions, weighing approximately 35 g, in order to test the representativeness of field sampling and to verify if the sample was collected from the same sediment layer. For every set of sample the same workflow was followed (*Fig. 3*).

Samples were dried on 105°C, gently crushed, homogenised and dry sieved at a 2 mm mesh size for removing larger clasts and organic constituents. The fraction below 2 mm was analysed with a Fritsch Analysette 22 MicroTec laser diffraction equipment with a 0.08-2000 μm measurement range and 108 measurement channels. Instrumental settings and protocols described by Kun et al. (2013) were used throughout the measurement process. Analyses were made in 3 steps for each parallel set of samples (Fig. 3). Firstly, the original untreated subsamples were analysed (Step 1). Subsequently, samples were treated with 10 % H_2O_2 for 1 day and a second run of measurements was performed after drying (Step 2). Finally, after a 1 day long 10 % HCl treatment a third run was also executed (Step 3). Acid treatment was aimed to ensure the complete removal of organic material, carbonates and to minimise the presence of aggregates.

Consequently, each sample was measured 6 times in all, the different measurements were identified by adding suffixes marking the set of samples and the steps of measurements (Fig. 3).

In the beginning of measurements the efficiency of ultrasonic treatment, made within the wet dispersion unit of the measurement device, was tested on 3 clayey, untreated samples, prone to be affected by aggregation. Ultrasonification is very efficient in removing clay coatings, but it can also brake up quartz grains if the exposure is too long (Di Stefano et al., 2010). Three sequential measurements were made, each preceded by 12s of treatment, then the results were compared.

Raw grain size data were exported and processed by software Gradistat v8. Grain size classes were identified following the Udden-Wentworth scale (Udden, 1914; Wentworth, 1922). For comparing different sets of samples and different steps of measurements cumulative distribution and the median diameter (D50) were primarily considered.

Textural properties were compared using the graphical method and the triangular diagram of Folk (1954) and Folk and Ward (1957). Subsequently the mean D50 value of different sample groups were analysed in order to reveal general tendencies and differences related to parallel sampling and sample pretreatment. Results were also compared on the level of major grain size fractions (clay, silt, sand). Finally, D50 data of different measurements were plotted against each other and correlation coefficients (R^2) were calculated in relation to a 1:1 linear function in order to determine the variability of the data and to provide further insight to factors modifying the measurement results.
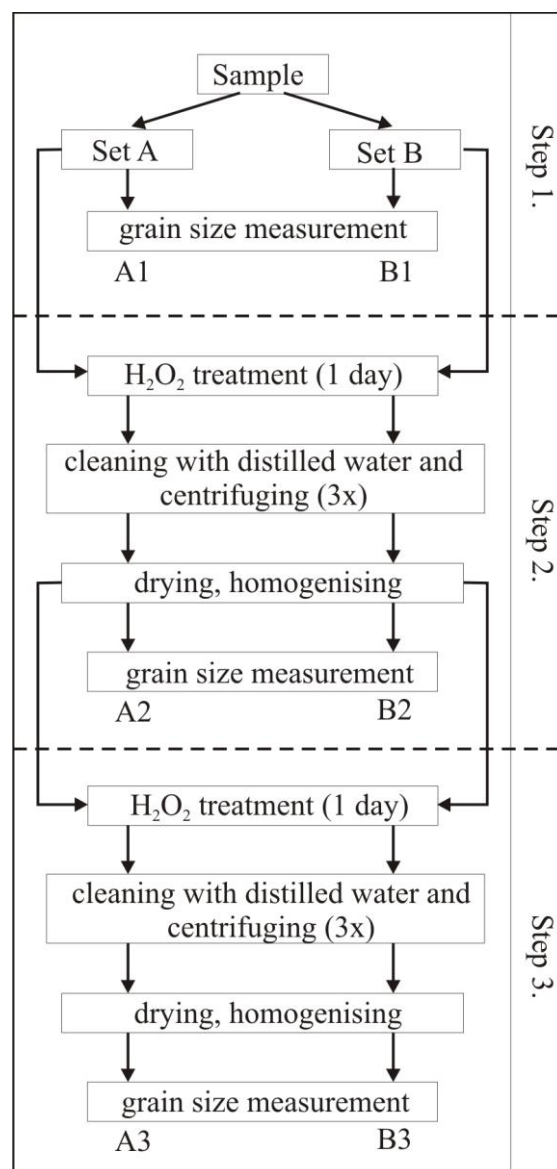

Fig.3 The steps of the measurement process and the identification of the different group of samples compared in the study

RESULTS AND DISCUSSION

Ultrasonic pretreatment

Regarding the median diameter of samples the mean relative difference between the first and third measurement cycles was 1.6 %, while maximum deviance was 3.3% (Fig. 4). Results were similar to those of Kun et al.
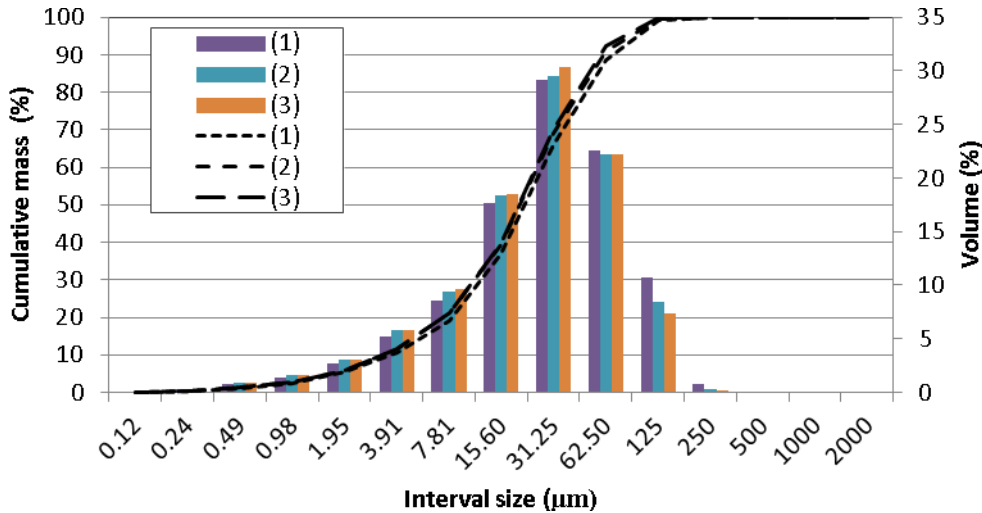
Fig. 4 Cumulative (dashed lines) and frequency (columns) particle size distribution of sample C4, using an increasing length of ultrasonic dispersion (1): 12s; (2): 24 s, (3) 36 s

(2013), using the same equipment, however, also corresponded well to the observations of Di Stefano et al. (2010), applying a longer ultrasound treatment. Based on the above, the data of the third measurement cycle, preceded by a total 36s of ultrasonification, were used for further comparisons.

It is assumed therefore that the applied treatment was adequate for the dispersion of clay aggregates and considering the results of Chappell (1998) the breaking up of individual grains could be also avoided.

Textural properties and main fractions

Based on the measurements on untreated samples, all belonged to two textural groups: sandy mud and muddy sand (Fig. 5.), representing 56 and 44% of the samples in case of Set A and 59 and 41% in case of Set B, respectively. If Step 2 and Step 3 results are taken a clear textural shift, i.e. fining due to disintegration can be noticed. As a matter of H_2O_2 treatment in case of both sets the proportion of the coarsest samples decrease by around 20%, and a new, finer textural group, mud also appears. Following HCl treatment fining is still remarkable on a textural level, and finally 30 and 24% of samples from Set A and Set B can be described as mud, respectively. Nevertheless, this time fining mostly affects sandy muds, and the proportion of muddy sands hardly changes.

These trends are reinforced if results are compared concerning the main fractions, being very similar at both sets of samples throughout the whole measurement process (Fig. 6). Fining is evident in this case too: the proportion of sand continuously decreases while the proportion of silt increases, and the proportion of clay first increases then remains stable. It is also obvious already at this stage of the comparison that as pretreatment advances the difference between the results of set A and set B samples is increasing (Fig. 6). These findings are in accordance with the results of Kun et al. (2013) and Di Stefano et al. (2010).
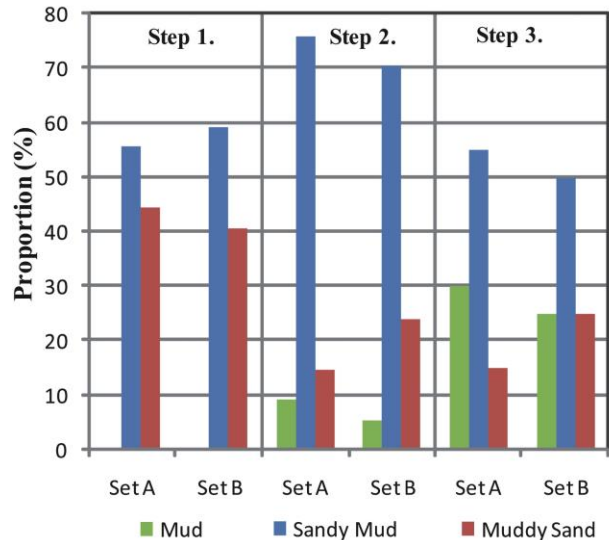

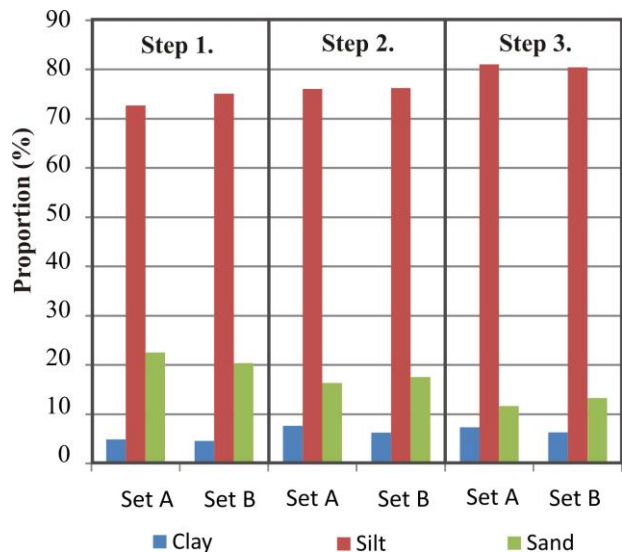Fig. 5 Textural classification of samples at different steps of the analysis

Fig. 6 Mean proportion of main fractions (clay, silt, sand) in both sets of samples at the different steps of the analysis

Consequently, acid pretreatment can significantly change even the textural properties of samples. At this stage of the analysis it is assumed that the removing of organic constituents rather affects the classification of coarser samples, while removing carbonates rather influences finer samples.

Nevertheless, it must be noted that textural shift is mainly because many of the samples are situated on the threshold between textural classes. In general the textural properties of the two parallel sample sets remained very similar throughout the measurement process.

Percentage deviations in median grain size

Concerning the entire dataset mean difference between the D50 value of A1 and B1 samples is 6.8% (0.53μm) in average. By pretreatment these values increase considerably and reach 23.7% (5.9μm) in case of A2 and B2 samples, while concerning A3 and B3 samples it drops back to 7.8% (1.9μm).

Differences are greater if the same set of samples are compared but with different pretreatment. For example in case of A1 and A2 samples the difference in D50 values is 33.7% (9.7μm) in average, while the same data for A1 and A3 samples are 14.3% (4.1μm). Concerning the two acid treatment steps it seems as if samples were more sensitive for H_2O_2 (A1-A2 and B1-B2), showing a 33.7% and a 14.6% difference than for HCl etching (A2-A3 and B2-B3): 22.7% and 9.3%. This emphasizes again the significance of acid pretreatment in changing the measured grain size distribution.

If results are separated on the basis of morphology, in the case of ploughing blocks and turf-banked lobes the average difference between A1 and B1 samples is 0.5% and 8.4%, respectively. However, after pretreatment the two groups swap, and the difference between A3 and B3 samples changes to 36% and 2.3% (Table 2). Thus, in the case of ploughing blocks acid treatment ruined the coherence of the results, while in case of turf-banked lobes it improved significantly. It has to be resolved in the future if there is any genetic explanation to this phenomenon: higher organic matter or carbonate content, or greater spatial variability in grain size composition for example.

When the main fractions are considered on their own, obviously the situation gets slightly better. Concerning raw samples (A1 and B1) differences remain below 3%. As a result of acid treatment the greatest difference is experienced in the case of the clay fraction (Table 2), being 23.4% and 15.8%. By the end of the measurement cycle the proportion of silt proved to be the most stable (3.7%) when the two sets of samples (A3 and B3) are taken.

A similar relationship is seen if the different steps of measurements are compared within the same set of sample, namely the highest variability can be attributed to the clay fraction (42.1% and 32.2%), while silt provides the most steady data if the raw and fully treated samples (A1-A3 and B1-B3) are compared. However, if the two steps of treatment are considered separately (A1-A2 and A2-A3 for example) it seems as if the sand fraction was less sensitive to acid pretreatment. The discrepancy is probably because the change in the sand fraction (decreasing abundance) is unidirectional at each step of treatment, while in the case of silt relative loss (silt particles turning into clay) and relative gain (sand particles turning into silt) can also occur, making the final result more comparable to the raw data. Finally, in general it seems as if samples, with the exception of the sand fraction, were more sensitive for H_2O_2 treatment (A1-A2 and B1-B2) than for HCl treatment (A2-A3 and B2-B3).

Correlation analysis

In order to check the consistency of comparative results correlation coefficients were calculated by plotting against the results of parallel measurements. Values of R^2 supported and also supplemented the conclusions made on the basis of mean percentage deviations (Table 3).

If the full set of samples is considered, then R^2 is the highest between untreated A1 and B1 samples (0.71) and as pretreatment went on its value significantly decreased (Table 3). If compared to changes in mean differences it must be noted that concerning the A3-B3 pair lower mean difference is not followed by the increase of the R^2 value, i.e. HCl treatment did not improve the comparability of the samples in the end (Table 3). It is also noteworthy that the correlation de-

Table 2 Mean percentage deviation of median diameter (D50) between different sample groups

% difference	median diameter (D50)			main fractions		
	all	blocks	lobes	clay	silt	sand
A1-B1	6.8	0.5	8.4	1.4	2.9	0.8
A2-B2	23.7	36.2	5.8	23.4	16.0	1.3
A3-B3	7.8	11.2	2.3	15.8	3.7	6.8
A1-A2	33.7	40.9	28.1	43.6	25.0	8.6
A2-A3	22.7	9.5	0.3	2.6	22.8	13.6
A1-A3	14.3	34.7	27.9	42.1	2.9	21.1
B1-B2	14.6	6.9	16.7	27.5	13.3	6.7
B2-B3	9.1	20.6	7.7	6.5	4.6	8.5
B1-B3	22.4	26.1	23.1	32.2	9.1	14.6

Notes: set A and B: untreated (step1: A1 and B1), pretreated with H_2O_2 (step2: A2 and B2) and with HCl (step3: A3 and B3)

Table 3 Correlation coefficients of median diameter (D50) between different sample groups

R^2	median diameter (D50)			main fractions		
	all	blocks	lobes	clay	silt	sand
A1-B1	0.71	0.93	0.69	0.96	0.94	0.82
A2-B2	0.49	0.24	0.72	0.80	0.47	0.60
A3-B3	0.42	0.37	0.78	0.87	0.72	0.47
A1-A2	0.81	0.22	0.86	0.75	0.32	0.63
A2-A3	0.39	0.34	0.37	0.70	0.24	0.16
A1-A3	0.26	0.12	0.17	0.62	0.28	0.08
B1-B2	0.46	0.62	0.50	0.77	0.57	0.56
B2-B3	0.80	0.93	0.92	0.77	0.50	0.09
B1-B3	0.60	0.70	0.61	0.55	0.23	0.08

Notes: set A and B: untreated (step1: A1 and B1), pretreated with H_2O_2 (step2: A2 and B2) and with HCl (step3: A3 and B3)

creased mostly after H_2O_2 treatment, the subsequent HCl etching just slightly affected the comparability of the samples.

When correlations within the same set of samples are taken, the values of the two sample groups are significantly different. In the case of Set A samples H_2O_2 treatment influences much less R^2 values compared to Set B samples, conversely, the HCl step introduces a much greater discrepancy (lower R^2) in case of Set A samples than the other group. If raw and fully treated samples are considered (A1-A3 and B1-B3) the deviation in correlation coefficients is also striking (Table 3), which might mean either that the mineral composition of subsamples was different, or acid treatment was not entirely consistent, however the same procedures were applied in each case.

The discrepancy above can be further analysed if the different solifluction forms are considered separately. Similarly to percentage deviations in median diameter ploughing blocks show a very good comparability on the level of the A1-B1 pair, which drops abruptly after the H_2O_2 step (A2-B2) (Table 3). In case of turf-banked lobes R^2 values are very similar throughout the whole process, referring to more uniform mineral composition. If the two sets of samples are considered separately, a great variation can be seen in the effects of pretreatment, just as in case of percentage deviations described above.

Correlations were calculated for the main fractions as well. Highest values were received for clay (0.96) and silt (0.94), for sand the R^2 value was somewhat lower (0.82) (Table 3). In case of the clay fraction correlation coefficients between different sets of samples remained reasonably high throughout the whole analysis, which seemingly contradicts the trend experienced for percentage deviations (Table 2). This might be because a slight change in median diameter can cause a significant difference in percentage deviations, while the R^2 value is less sensitive to this effect. Based on the coefficients, the variability in the silt fraction increases significantly after H_2O_2 treatment, which is in harmony with the results received for percentage devia-

tions. Although R^2 values received for the sand fraction of untreated samples (A1-B1) is reasonably high, with the advance of pretreatment the largest variability is introduced by far here. Actually, in the end, when results are plotted against within the same subsample group, no functional relationship can be identified (Table 3). This phenomenon is primarily due to the disintegration of particles as a result of HCl etching (A2-A3 and B2-B3).

CONCLUSIONS

In the present paper we investigated the representativeness of sampling in case of different solifluctional landforms, and the effect of acid pretreatment on LD grain size measurements.

If the textural classification and fractional composition of subsamples is considered, the results show a high consistency between the two parallel measurements let they be made on untreated or pretreated samples. An overall fining as a matter of etching is evident. Based on the experienced shifts between textural classes, fining as a result of H_2O_2 treatment is a greater issue in case of muddy sands, while fining as a result of HCl treatment is rather significant in case of sandy muds. This implies a compositional difference between samples falling to coarser and finer textural groups.

Either considering mean percentage deviations or correlation coefficients the comparability of the parallel measurements is best if samples remain untreated (A1-B1). In this sense the sampling strategy in general is validated, however considering different landforms clear differences were experienced. While parallel untreated samples yielded very similar results in case of ploughing blocks, in case of turf-bank lobes a more careful and detailed sampling is proposed for further geomorphological comparisons, as probably large samples include more than one structural or stratigraphic elements of the landform.

In case of the present samples pretreatment introduces a major uncertainty to further comparison and interpretation. In general relative deviation increases

and correlation decreases as pretreatment advances. Based on the analyses, HCl etching results a greater deviation and variability in case of the sand fraction, in turn H_2O_2 rather affects the silt fraction. This can partly be traced back to the geological background and composition of samples, namely the greatest deviations are experienced in case of landforms developed on crystalline limestone, and finer fractions are more likely to contain organic constituents of their size range.

Nevertheless, in several cases Set A and Set B samples exhibited different tendencies during the pretreatment process. This might imply either that organic and carbonate content could be different at parts of the relatively large samples, or the etching process was not entirely consistent. Both possible reasons require further analysis. High variability of organic content can be explained by the stratigraphic observations of Hugenholtz and Lewkowicz (2002), Kinnard and Lewkowicz (2006), Oliva et al. (2009), who revealed buried organic horizons, overlapping and deformed layers in solifluctional forms. Consequently, organic matter and carbonate content determination as well as additioal mineralogical analyses can add further insight to the interpretation of discrepancies. Meanwhile, by changing the parameters of acid pretreatment and making further comparative measurmeents the methodology of etching can be refined.

Finally, based on the results of the above research, we advise to make always multiple LD measurements on a representative group of samples from the entire sample set before the geomorphological or environmental interpretation of results. This way both sampling and sample processing can be validated, and the uncertainties of conclusions can be decreased.

Acknowledgements

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