VERSITA

GEOCHRONOMETRIA 30 (2008), pp 41-47
DOI 10.2478/v10003-008-0005-x

Available online at
versita.metapress.com and www.geochronometria.pl

A SIMPLE METHOD TO SEPARATE QUARTZ AND FELDSPAR AND ITS APPLICATION TO TL/OSL METHODS

DANIEL FERNÁNDEZ MOSQUERA and JORGE SANJURJO SÁNCHEZ
Instituto Universitario de Xeoloxía "Isidro Parga Pondal", Universidade da Coruña, Campus de Elviña s/n. 15071. A Coruña. Spain

Received 4 April 2007 Accepted 30 October 2007

Abstract: In the dating of sediments by luminescence methods, to obtain a sample the purest possible as to its mineralogy determines the rest of the analytical procedures. The isolation of the sample minerals is a critical stage due to the impossibility of quantifying the relative contributions to the dose from the different minerals. In this paper we propose a non chemical method to isolate quartz from feldspar extracted from different aeolian and fluvial sediments. Grinding together quartz and feldspar grains in an agate mortar results in crushing the feldspar fraction due to its smaller hardness. Later sieving of the grinded mixture enables simple separation of both fractions. Then it is possible to save the feldspar fraction for later analyses instead of losing it as it occurs when using acid etching. Also, we study the evolution of the feldspar abundance along different purification steps with acid etching or grinding with two numerical indexes. We conclude that the grinding procedure can be introduced in the purification methods as an effective way to separate quartz from feldspar. Nonetheless, it is not an standalone procedure to obtain pure quartz and must be combined with chemical methods when the sample is rich in feldspar minerals.

Keywords: quartz dating, feldspar dating, OSL, IRSL.

1. INTRODUCTION

The dating of sediment and geological events is one of the main applications of the luminescence method. The measurement of the palaeodose (i.e. environmental radiation dose absorbed by the mineral) is the basis of the Thermoluminescence (TL), Optically stimulated Luminescence (OSL) and Infra Red Stimulated Luminescence (IRSL) methods (Aitken, 1985 and 1998). The particular features of this paleodose, especially as to stability and intensity of the signal, belong to the mineral crystalline structure. Thus, different minerals, though having shared the same environmental conditions of radiation, store and free the energy of ionizing radiation in a different way. In the luminescence measurement, the characterization of the luminescent contribution of each mineral may not be even quantified. Therefore, it is necessary to isolate the mineral before the luminescence measurements.

Among the minerals with luminescent properties, quartz and feldspar are the most used ones. Both have advantages and disadvantages making them complementary for sediment dating purposes. Nevertheless, as the luminescent signal stored on the quartz is not affected by anomalous fading, it is usually preferred over the feldspar for sediment optical dating. With respect to the excitation source, quartz shows luminescence emission during OSL and TL measurements, however, very weakly under IRSL stimulation; feldspars show emission under all three kinds of stimulation. The thermoluminescence signal may be assigned to quartz or feldspars because those minerals differ in temperatures at which emission occurs. The IRSL signal is almost exclusively observed in feldspars (expect high temperature stimulation); quartz and feldspars show OSL emission, though feldspars emission is usually brighter.

The sample preparation before the luminescence measurement has two goals: mineral isolation and removal of the alpha particles contribution to the total dose. In general, the sample undergoes etching with HCl to eliminate carbonates that emit spurious signal in OSL and TL, and treatment with H_2O_2 to eliminate organic matter. For the mineral isolation, commonly chemical etching is used with the application of a silicate solving acid (HF or

Corresponding author: D. Fernández Mosquera
e-mail: xemos@udc.es

ISSN 1897-1695 (online), 1733-8387 (print)

H_2FSi_6) or/and the density separation. The latter is used when the purpose is to analyse the feldspar fraction, and is a rather skilled and time consuming procedure. The acid etching aims at preferentially dissolving feldspars, even if some quartz is lost in the process, and also to remove the mineral outer layer which stores the alpha radiation contribution to the luminescence signal. In both methods valuable sample is lost, which can be a problem if the original sample is scarce. Finally, it is fundamental to verify the success of the isolation. Up to now, the most complete methodology is the one described in Mauz and Lang (2004). The authors compare four different indexes to establish the presence of feldspars in the sample. Also, they show that the etching with concentrated HF during a short time is very effective in dissolving feldspars.

In this paper, we propose a non chemical method to isolate quartz from feldspar. The procedure is based on the difference in hardness of both minerals. The quartz hardness is between 7-7.5 on the Mohs' scale, while the feldspar hardness is normally lower (6-7). The friction between quartz and feldspar should result in breaking up feldspar grains into particles of smaller size, while leaving quartz unaltered. Therefore, sieving of a mixture of quartz and feldspars after having undergone grinding would result in a higher presence of the feldspar grains in the smaller fraction and of quartz grains in the larger one. This method would enable the isolation of both minerals without losing them. The advantage of this method, apart from being relatively non destructive, is that it is very simple and quick, environmentally friendly, it saves reagents and expensive equipment is not necessary.

2. MATERIAL AND METHODS

In order to prove the result of the procedure for different proportions of quartz and feldspars, two types of samples were selected: aeolian samples with low feldspar content and fluvial samples with a larger feldspar content.

The aeolian samples are from the NW of the Iberian Peninsula. They are sediments with grain size between 0.35 mm and 0.18 mm for 85% of each sample. Mineralogically it is composed almost totally of quartz and the feldspar fraction is only detectable through luminescence and not by X-Ray Diffraction. The fluvial samples are sediments of the Euphrates River (Syria) and have a larger proportion of feldspars, easily seen on optical inspection.

The mortar grinding procedure consists of exerting a slight pressure on the sample against the mortar wall, balancing the pestle of the mortar during about 3 seconds. An agate mortar is employed because its hardness lies between the quartz and feldspar ones. To verify whether the pressure exerted by the mortar on the quartz does not induce triboluminescence or alteration of the accumulated dose measurable by OSL a sample of known palaeodose was needed. We used a sample of irradiated (5 Gy) quartz provided by Risø because its low palaeodose makes it more sensible to any alteration. OSL measurements of five aliquots before and after mortar grinding showed undistinguishable results. Thus, we can assume that no measurable perturbation of the dose is introduced by this method when the quartz paleodose is equal or larger than 5 Gy.

In the case of aeolian samples, 4 aliquots of each sample (A, B, C and D) were separated in order to test several combinations of chemical and mechanical purification steps. The mortar grinding in these samples consisted of a single step. Then, they were sieved to isolate the fractions <0.180 mm (S) and >0.180 mm (L). In each fraction the feldspar abundance was measured according to the indexes proposed by Mauz and Lang (2004). The diagram of the different treatments the aliquots were subjected to can be seen in **Fig. 1**. The rectangles indicate a chemical or mechanical treatment; the rhombus, an abundance measurement of feldspars by luminescence, to which a code is assigned to represent the results of that measurement in the graphs; the semi-circles indicate a sieving step to separate fractions greater or smaller than 0.180 mm after mortar etching.

The measurements made were the following:

1. Measurements of the feldspar abundance (WF-1) in the original sample, with grain diameter ranging between 0.09-0.3 mm, and after undergoing a standard chemical purification to dissolve carbonates and organic matter (WF-2).
2. Separation of four aliquots: A, B, C, D. Each one undergoes a different combination of chemical and mechanical etching steps.
3. Aliquot A:
    1. Feldspar abundance measurement in the sample after chemical etching with HF (10%) (A-1).
    2. Mortar grinding and isolation of fractions larger (L) and smaller (S) than 0.180 mm. Feldspar abundance measurement in the larger (AL-2) and smaller (AS-2) fractions.
4. Aliquot B:
    1. Mortar grinding and isolation of fractions. Feldspar abundance measurement in the larger (BL-1) and smaller (BS-1) fractions.
    2. Etching with HF (10%) on both fractions (BL-1 and BS-1). Feldspar abundance measurement after the etching (BL-2 and BS-2).
5. Aliquot C:
    1. Mortar grinding and isolation of fractions. Etching with concentrated HF (40%) on both fractions. Feldspar abundance measurement in the larger (CL-1) and smaller (CS-1) fractions.
6. Aliquot D:
    1. Feldspar abundance measurement in the sample after a chemical etching with concentrated HF (40%) (D-1).
    2. Mortar grinding and isolation of fractions greater (L) and smaller (S) than 0.180 mm. Feldspar abundance measurement in the larger (DL-2) and smaller (DS-2) fractions.

In the samples with higher feldspar content (fluvial sediments of the Euphrates River), we decided to apply some previous acid etching before measuring feldspar abundance indexes (see **Fig. 2**). Previous acid etching of the sample would allow us to start from a lower feldspar abundance suitable for comparison with the aeolian ones and large enough for testing the efficiency of the mortar

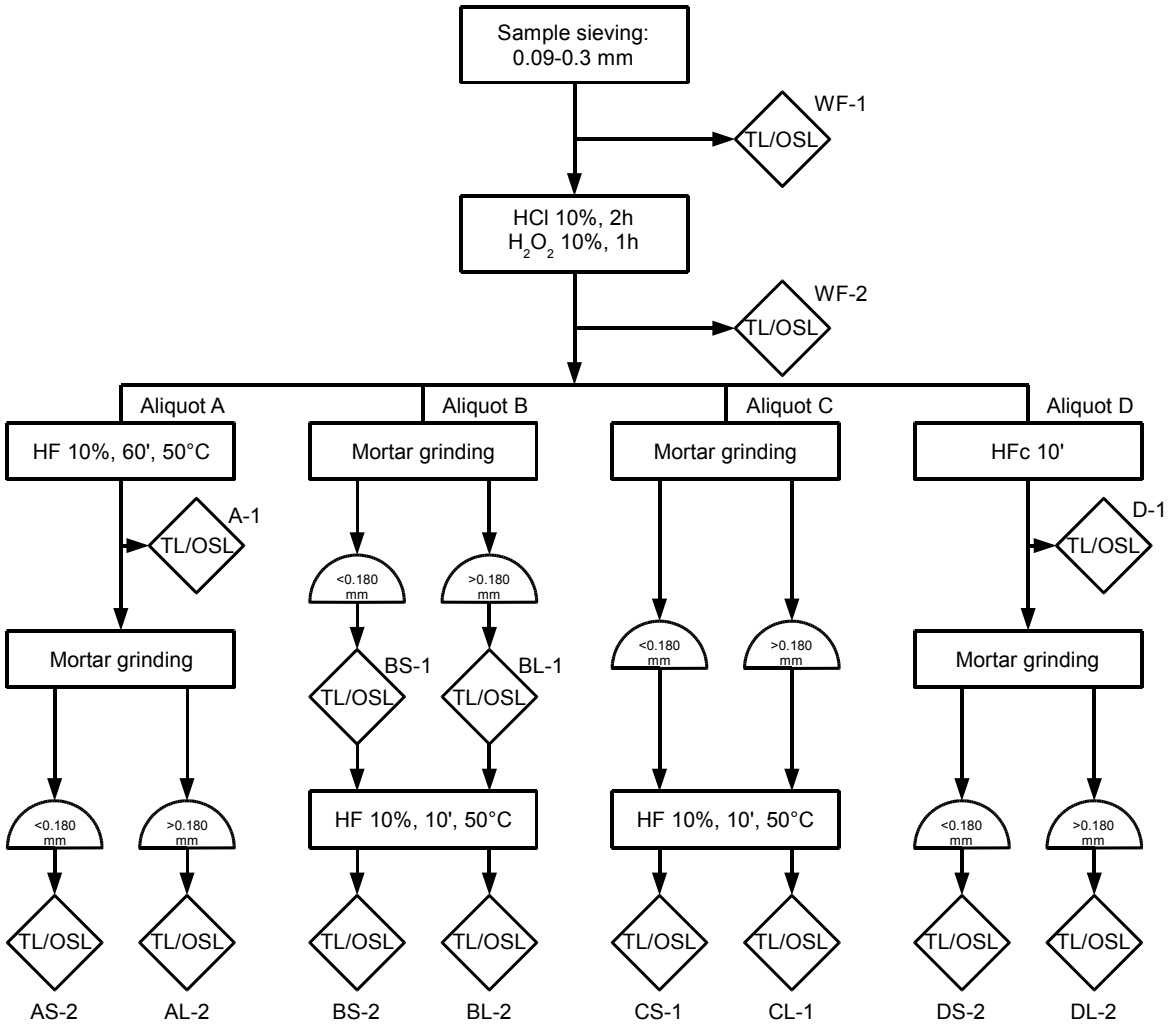

Fig. 1. *Scheme of the sequence of chemical and mechanical treatments that the aeolian samples underwent. The mortar step consists of a single grinding of 3 seconds followed by sieving. Each measurement of feldspar abundance is identified by a code standing for aliquot-fraction-measurement cardinal number.*

grinding. This sample's natural feldspar content would make the mortar grinding procedure and endless step if we wanted to reach the same levels of quartz purity than aeolian samples.

After the chemical etching with HCl (10%) and H_2O_2 (10%) to dissolve carbonates and organic matter, two fractions were separated: one between 0.09 and 0.180 mm (S) and another between 0.180 and 0.250 mm (L). Each of these fractions underwent two consecutive digestions with HF progressively concentrated. The etchings were carried out at 50ºC to enhance reaction rate, although this could induce thermal transfer effects on the feldspars (Wallinga *et al.*, 2001). If the intention is to use the separated feldspar fraction for further analyses, the etching should be done at room temperature. Between the two digestions, the fractions were crushed in the mortar and were sieved to keep the fractions >0.090 mm (S) and >0.180 mm (L). After the second etching with HF the feldspar abundance was measured in each fraction (S1 and L1), taken as starting abundance for the comparison of the mortar effect as single separation and purification method. On this occasion, several mortar steps were made before a new measurement. After each step, made as the one described for the aeolian samples, the samples were sieved to discard the smaller fraction, <0.180 mm in fraction L or <0.090 mm in fraction S, which was weighted. Thus, several measurements were done on each fraction. In the >0.180 mm fraction (L): L2, after 8 mortar steps; L3, after 4 mortar steps (12 accumulated); and L4, after 6 steps (18 accumulated). In the >0.09 mm fraction (S): S2, after 25 mortar steps and S3, after 35 steps (60 accumulated).

For inferring feldspar content on the treated samples, two indexes were calculated from OSL, TL and IRSL measurements on the samples. The TL/TL index was calculated following the procedures described by Mauz and Lang (2004). TL/TL is the ratio between TL at 220ºC (integral from 200 to 250ºC) and TL at 110ºC (integral from 90ºC to 140ºC). The IRSL/OSL is a modification of the IRSL index proposed by Mauz and Lang (2004). This index is the ratio between the IRSL signal (integral between 0 s and 10 s illumination time, after subtraction of the background count rate from the 60-100 s interval) and the OSL signal (integral from 0 s to 5 s, after subtraction of the background count rate from 25 s to 40 s).

Two aliquots of each sample were mounted on Al discs using silicone spray. The measurement sequence used is the one described by Mauz and Lang (2004). For the TL/OSL/IRSL measurements, a Risø reader model TL/OSL-DA-15 was used equipped with an EMI 9635 QA photomultiplier tube. Optical stimulation was provided by Nichia blue light-emitting diode arrays providing 38 mW (100% power at 470 nm) and Vishay IR LED's delivering 133 mW at 870 nm (100% power). The UV emission band was isolated using a Hoya U340 filter. The reader is equipped with a $^{90}Sr/^{90}Y$ beta source which provided 0.140±0.003 Gy/s during all measurements. The heating rate was 5ºC/s in all the measurements.

3. RESULTS AND DISCUSSION

The purpose of this study is to verify the possibility of isolation of quartz from a mixture of quartz and feldspar without loosing significant amounts of sample in sediments of common use in luminescence dating. The use of the agate mortar should break up feldspar grains when rubbing them against the quartz due to their lower hardness. Therefore, sieving after the grinding should separate smaller feldspar grains into the thin fraction, leaving the thicker one enriched in quartz grains. Another question is which is the most adequate procedure to carry out this isolation and to establish what is its efficacy in comparison to chemical purification methods. Finally, we discuss which requirements does the sample have to fulfil for this method to be effective and how to use it.

The evaluation of these purposes depends on the method that we select to measure the presence of feldspars in the sample. Mauz and Lang (2004) propose up to 4 different indexes, concluding that there is not any standing alone index. They also suggest that the numeric indexes should be combined with the luminescence graphs to obtain better results. Therefore, the said indexes cannot define an absolute scale that allows us to compare one sample with others. In our estimation, we have decided to use only two indexes, the TL/TL and the ratio IRSL/OSL. The choice of this two indexes relies on that each one measures a specific feature of the feldspar minerals (TL emission at 220ºC and IRSL emission), so they can be considered as complementary (Mauz and Lang, 2004). The ratio IRSL/OSL normalizes the feldspar IRSL signal to the quartz OSL signal, analogously to the TL/TL index, becoming more comparable. In both indexes, the higher ratios indicate greater presence of feldspars. As the indexes are not absolute, there is no maximum value for them. If there were no feldspar, their values would be very close to zero, perhaps without reaching it due to the weak quartz emission when stimulating it with IR light. So, we think it is more adequate to study how these indexes evolve during the purification process.

The aeolian samples, poor in feldspars, are an adequate sample for evaluating the mortar effect as isolation method among the mineral phases. In **Figs 3a** and **3b** there are presented the TL/TL and IRSL/OSL indexes for each step of the analytical procedure, coded in the **Fig. 1**. We have divided the measurements in three groups for making data evaluation simpler. In general, more sensitivity to feldspar abundance can be inferred from larger

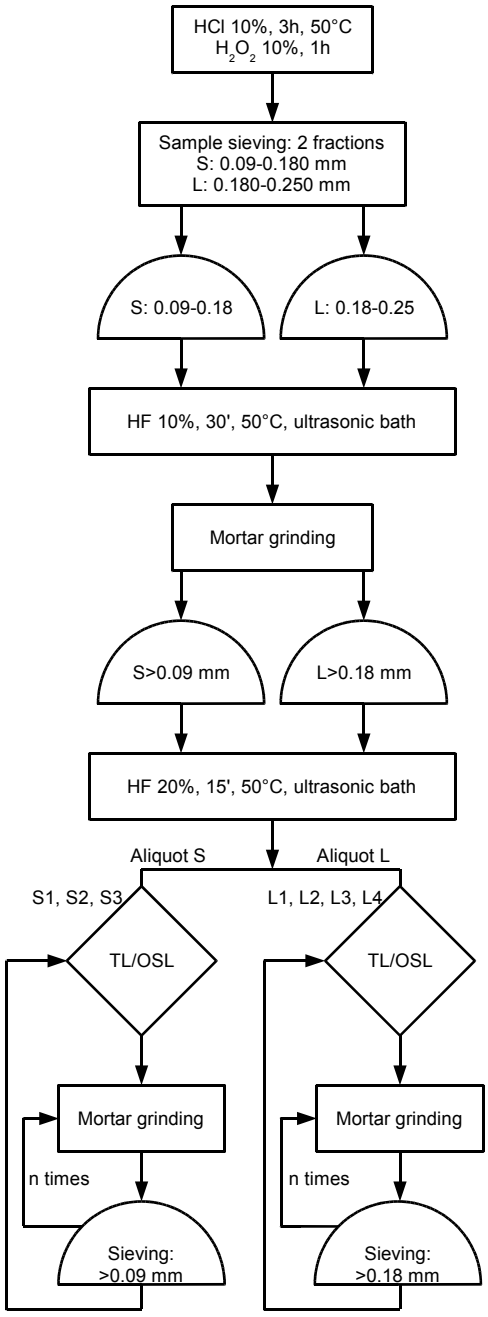

Fig. 2. *Scheme of the sequence of chemical and mechanical treatments that the fluvial samples underwent. Each measurement of feldspar abundance is identified by a code standing for aliquot-fraction-measurement cardinal number. The number of times of each mortar/grinding/sieving sequence is stated in the Material and Methods section.*

IRSL/OSL than TL/TL values, although both indexes reflect the same patterns.

The first group (measurements WF-1, WF-2, A-1, D-1, BL-1 and BS-1) is made to compare the effect of the mortar grinding procedure (BS-1 and BL-1) with the sample unaffected by any feldspar purification method (WF-1 and WF-2) and the sample etched with HF (A-1 and D-1). Step BS-1 shows group largest values on both indexes, which means that feldspar effectively concentrates on the smaller fraction after a single grinding step. This does not imply an equal drop on the BL-1 values, as

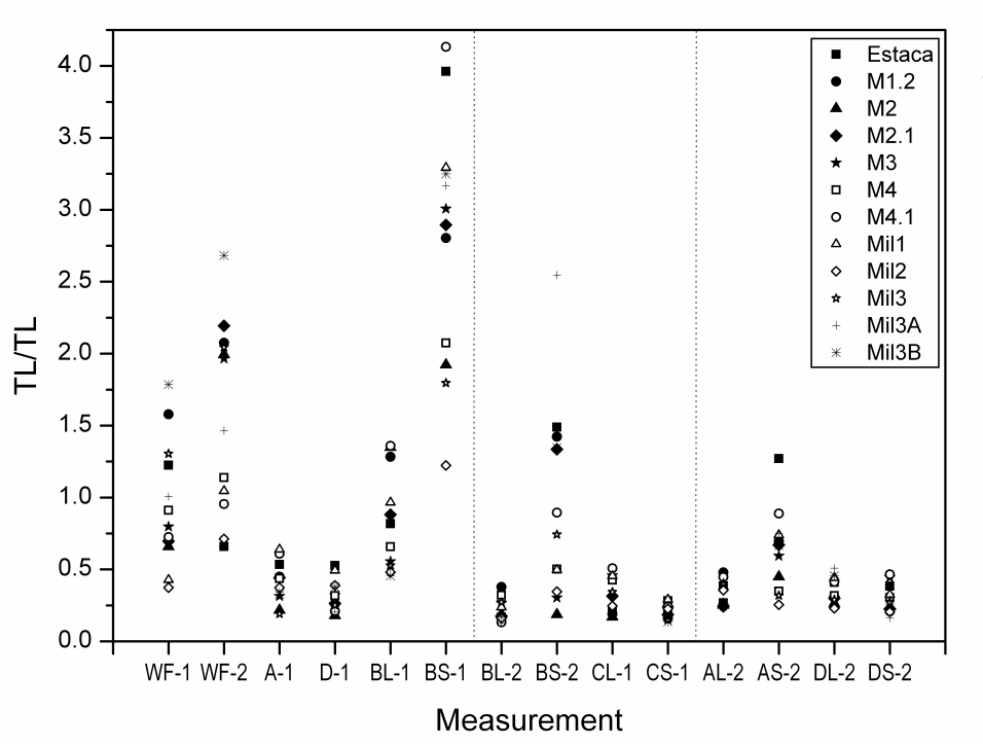

Fig. 3a. *Aeolian samples TL/TL index. Measurement axis refers to the analytical procedure scheme.*

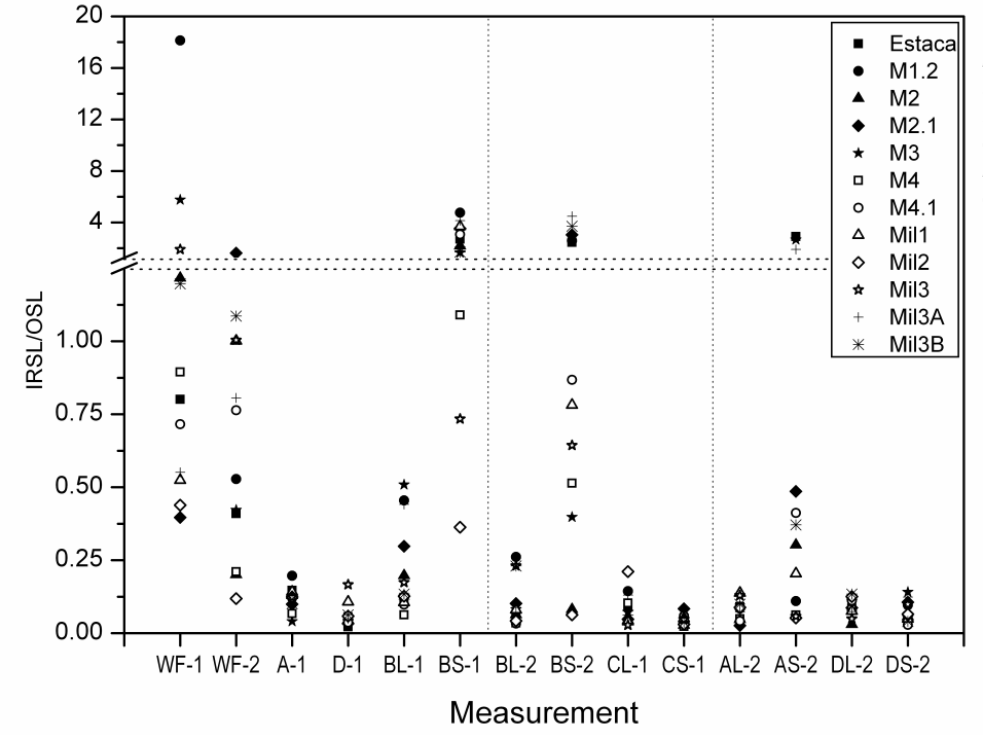

Fig. 3b. *Aeolian samples IRSL/OSL index. Measurement axis refers to the analytical procedure scheme. IRSL/OSL axis shows a break in order to display larger values and have a good resolution for lower ones as well.*

they remain similar to the unaffected sample (WF-1) ones. This could mean that only qualitative evaluation can be done with the indexes as we used them. Maybe a weight normalization of the indexes (S. Huot, personal communication) could improve quantitative information. The quartz enriched fraction after the grinding procedure (BL-1) has larger index values than the acid etched fractions (A-1 and D-1). This clearly implies that acid etching is more effective in reducing feldspar amount. Both diluted and concentrated acid reach the same index figures, suggesting that for low feldspar content samples the acid concentration is not of critical importance for good purification.

The second group (BL-2, BS-2, CL-1 and CS-1) and the third group (AL-2, AS-2, DL-2 and DS-2) are made to investigate whether grinding the sample before (second group) or after (third group) the acid treatment could make any difference in the purification process. Results

show lower and comparable values for all the steps where concentrated HF is involved, regardless of the size of the grains, and/or the purification is done on the large fraction independently of the sequence. When the purification is done on the smaller, enriched in feldspars, fraction only concentrated HF leads to low values (CS-1 and DS-2). The use of diluted HF and mortar grinding, regardless of the sequence, leads to larger values (BS-2 and AS-2). This would mean that when feldspar content increases, diluted HF is not as efficient as the concentrated one. Regarding the use of a single grinding step as a purification method, it can not be stated that it is an efficient method when compared to acid etching. However, it is still valuable for separating quartz from feldspars.

The obvious question that follows is what happens when using several grinding steps. First tests on aeolian samples did not show much improvement after grinding 3 times more, although large variability in inter-aliquot results did not allow reliable data evaluation. Then we need samples with larger feldspar abundance to test if more steps can make a real difference in the purification process. In the given fluvial samples, richer in feldspar than the aeolian ones, then we investigate if several consecutive mortar crushings would reduce the feldspars in the larger fractions significantly. The interest of this purpose is that the acid etching also dissolves quartz, thus repeated acid etchings, necessary when feldspar content is large, reduce the available quartz for analysis. For the sake of clarity of the graphs, we will only show the results of one (TAF) of the two analysed samples because they are identical. In this sample, we start with two different grain fractions, a larger one (L) ranging between 0.180 and 0.250 mm, typically used in aeolian samples, and a smaller one (S), ranging between 0.090 and 0.180 mm, used in fluvial samples due to a possible better bleaching. Both fractions underwent acid etchings and one mortar grinding in order to obtain a sample with less feldspar content than the natural one, but even then greater than aeolian samples one. In order to reduce measurements dispersion, we used five aliquots per sample instead of two as in the aeolian samples. The values of the represented indexes correspond to the mean of 5 aliquots, and the error bars are the standard deviations.

In **Figs 4** and **5,** we have represented on the left Y

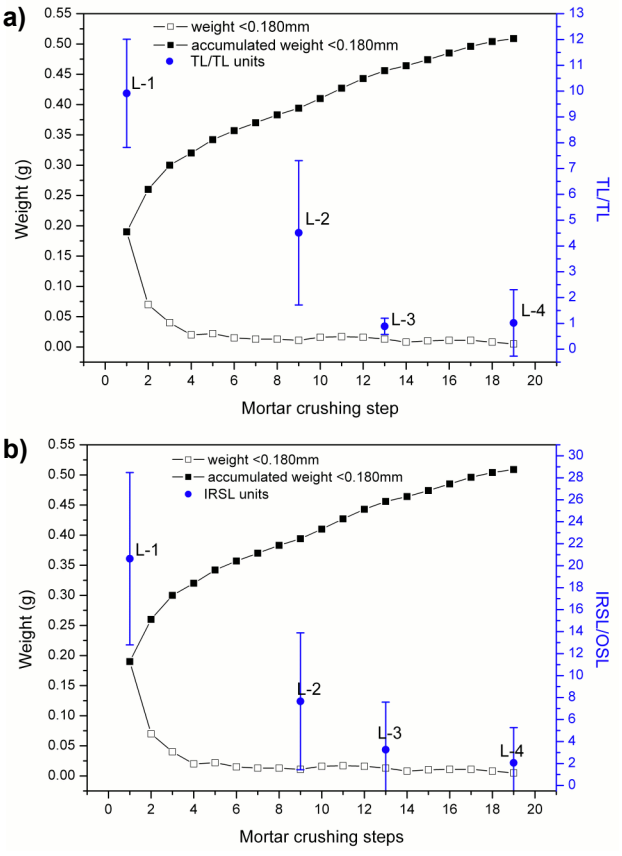

Fig. 4. *TL/TL, and IRSL/OSL indexes of fluvial sample TAF, 0.180-0.250 mm fraction (L). Likewise, this plot shows: the smaller fraction (<0.180 mm) weight obtained in each mortar crushing step and the accumulated weight along the procedure (left Y axis); the measured indexes at its precise mortar crushing step (right Y axis); and the crushing step (X axis). Indexes values correspond to the mean of 5 aliquots and error bars correspond to standard deviation. Note that IRSL/OSL index shows both much larger values and dispersion. However, after 19 crushing steps with no chemical etching in between, indexes show a significant reduction in feldspar abundance.*

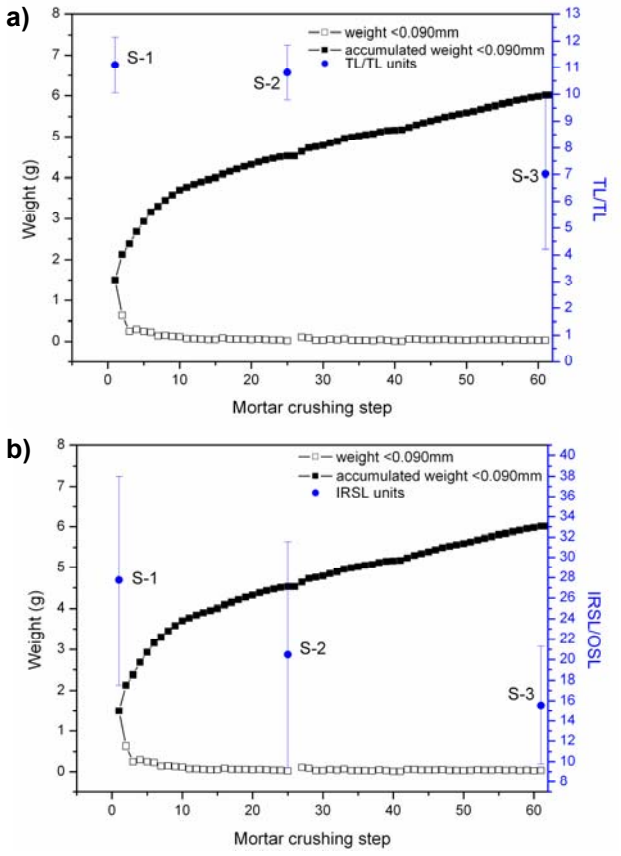

Fig. 5. *TL/TL, and IRSL/OSL indexes of fluvial sample TAF, 0.090-0.180 mm fraction (S). Likewise, this plot shows: the smaller fraction (<0.09 mm) weight obtained in each mortar crushing step and the accumulated weight along the procedure (left Y axis); the measured indexes at its precise mortar crushing step (right Y axis); and the crushing step (X axis). Indexes values correspond to the mean of 5 aliquots and error bars correspond to standard deviation. IRSL/OSL values show the same differences regarding absolute values and dispersion than the larger fraction. However, for this fraction feldspar reduction efficacy is smaller, even after many more crushing steps.*

axis the weights of the smaller fraction after sieving the sample ground by mortar. These weights correspond to each sieving and to the weight accumulated after n steps. In the larger fraction (L), the fraction <0.180 mm is weighed and discarded; the larger one (>0.180 mm) is collected and ground at the mortar again. This process is repeated n times and eventually, the large fraction is analysed to check feldspar abundance. Likewise, with the smaller fraction (S), the fraction <0.09 mm is weighed and discarded after sieving. The larger fractions (>0.180 mm and >0.09 mm) are measured to see how their feldspar content decreases, which is represented with the TL/TL, and IRSL/OSL indexes on the right axis of the graphs.

S-1 and L-1: This are the starting index values before the samples undergo the consecutive mortar grinding. The indexes show different and much higher values than in the aeolian samples. However, the index values are similar between the two fractions, S and L, indicating that at this moment the smaller fraction is not more abundant in feldspar. Both fractions underwent only one mortar step between the acid digestions.

L-2, L-3 and L-4 (see **Fig. 4**): With respect to the obtained weight of the finer fraction, that is, the feldspar-concentrated one, it is observed that the first steps isolate more quantity. After the sixth mortar step, the isolated quantity is practically constant in each case. Likely, the accumulated weight continues to increase, thus with each step we obtain more feldspar in the finer fraction. As to what regards the evolution of the indexes, it is observed that they decrease progressively down to the step number 13. Afterwards, there is little difference between the values of steps 13 and 19. The decreasing trend defined by L-1, L-2 and L-3 suggests that more than two or three grinding steps are necessary after the first one to make the difference clear, if we take into account the standard errors. The low values obtained using only the grinding, with no chemical etching, suggest that it is an effective method to separate quartz and feldspar when the latter is abundant in the sample.

S-2 and S-3 (see **Fig. 5**): the same pattern is observed with respect to the evolution of the isolated quantity in each case. The difference with the samples of greater grain size is that more mortar steps are needed to reduce the feldspar content of the larger fraction. In fact, after 60 steps, the indexes are even greater than the ones obtained with 19 steps in samples L. This indicates that rubbing is less effective when it is carried out with small size grains. Probably, the pressure exerted by the pestle on the grains diminishes as grains move more fluidly than the larger ones. However, after 60 grinding steps the decreasing trend defined by S-1, S-2 and S-3 suggests that even more steps could reduce the feldspar content.

4. CONCLUSIONS

The isolation of quartz and feldspar using the agate mortar has shown to be an effective method in samples with high and low feldspar content. In the samples with a low feldspar content, this method is not more advantageous than the HF acid etching to obtain a purified quartz fraction, instead it may be useful to concentrate the present feldspar and use it for IRSL measurement. Otherwise, in the samples with greater feldspar abundance, this method enables a significant reduction of the content in feldspar in the quartz fraction without using HF. The advantages of this procedure are simplicity, low cost and safety.

The evaluation of feldspar content by means of the indexes proposed by Mauz and Lang (2004) seems to be limited by sample homogeneity. The scarce quantity of sample that is measured by means of OSL is a determining factor of the obtained results, thus it is necessary to analyse several aliquots of the sample to obtain reliable results. The dispersion of each concrete index, it is greater in the IRSL/OSL index. This suggests that the IRSL/OSL index is more sensitive to detecting feldspar but not necessarily more reliable when evaluating the purification progress.

ACKNOWLEDGEMENTS

The authors wish to thank two anonymous reviewers for comments and criticisms which improved this paper. We also want to thank Ana Isabel Rodriguez Fraga for her laboratory work and Ana Martelli for English translation. Finally, DFM wants to thank to Kurt Marti for suggesting the method presented in this paper. This work was supported by a University of A Coruña grant for new research groups.

REFERENCES

Aitken MJ, 1985. *Thermoluminescence dating*. Academic Press, London: 267 pp.

Aitken MJ, 1998. *An introduction to optical dating*. Oxford University Press: 359 pp.

Mauz B and Lang A, 2004. Removal of the feldspar-derived luminescence component from polymineral fine silt samples for optical dating applications: evaluation of chemical treatment protocols and quality control procedures. *Ancient TL* 22(1): 1-8.

Wallinga J, Murray AS, Duller GAT and Törnqvist TE, 2001. Testing optically stimulated luminescence dating of sand-sized quartz and feldspar from fluvial deposits. *Earth and Planetary Science Letters* 193 (3-4): 617-630, DOI 10.1016/S0012-821X(01)00526-X.