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Re-examination of common extraction and purification methods of quartz and feldspar for luminescence dating

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Abstract

We tested common mineral extraction and purification procedures used in luminescence dating. Coarse grain quartz and alkali feldspar (KF) were extracted and etched from three samples of different geological setting and mineralogical maturity, using a variety of laboratory procedures. This was followed by particle size distribution measurement, SEM/EDS and XRF analyses, and D_e determinations employing SAR protocols – all used to characterize the mineral extracts.

Etching quartz in 40 % HF for 40 min removed all feldspars, with some preferential etching along fissures and grain boundaries. For mineralogically less mature samples, HF reduced average quartz grain size by 30–50 μm, whereas in mature samples extended etching of up to 60 min reduces grain size by ~10 μm. Ca-fluorides precipitated during HF etching but were fully removed by soaking overnight in 16 % HCl. Extracting quartz by heavy liquids or by magnetic separation (both followed by HF etching) resulted in equally pure quartz, with statistically identical D_e values.

Soaking KF for 40 min in 10 % HF resulted in uneven etching, severe micro-scale erosion along cleavage plains, grain disintegration, substantial precipitation of K-fluorides and the reduction of more than 100 μm in modal grain size, probably due to grain breakage along etched plains. The effects of etching vary greatly between samples from different geological provinces; more mature samples are etched in a more uniform way whereas mineralogically less mature samples are fragmented by etching.

The best benefits for KF are obtained by etching in diluted HF for short duration. For quartz, more concentrated HF and longer etching might be considered if grains are to have their entire α-affected zone (20 μm) removed. Since concentrated HF dissolves all feldspar contamination in quartz, there is no need for re-sieving after HF as that would bias the original grain size. Our results provide confidence in standard laboratory quartz sample preparation.

Keywords: Luminescence, HF-etching, K-feldspar, Sample preparation

1. Introduction

Extracting coarse (fine-sand size) quartz or alkali feldspars from sandy sediments for luminescence dating usually follows routine and established laboratory protocols (e.g. Wintle, 1997). This involves sieving, dissolving carbonates and oxidizing organic material, and concentrating quartz or feldspars using heavy liquids (density separation, Aitken, 1998, p. 67) or magnetic separation (Porat, 2006). To remove the α-affected zone of quartz grains, these are etched in concentrated hydrofluoric acid (HF) followed by soaking in hydrochloric acid (HCl) to dissolve fluorides. It was estimated that 40 min etching in 40–48 % HF would remove 10 μm from the outer zone of the grains – the area affected by α particles (Aitken, 1985, p. 255–258); this estimate was

based on weight loss during etching, since devices that measure particle size by laser diffraction were not available at the time.

The revival of alkali feldspar (KF) dating using the post IR-IR at elevated temperatures (Thomsen et al., 2008; Buylaert et al., 2009; Li & Li, 2011), brings again to attention issues concerning dose rate evaluations, namely the a-value and internal K-content of the extracted KF; knowing these correctly is crucial for obtaining accurate and precise ages. For a-values, a very large range can be found in the literature, from 0.2 for coarse grains (Rendell et al., 1993) to 0.07 for fine grains (Kreutzer et al., 2014). Removing the α-affected zone by etching would be most beneficial as knowing the a-value would no longer be necessary. Currently, laboratories vary in their practice, from not applying any etching at all (e.g., Porat et al., 2004; Trauerstein et al., 2014), to extended immersion in diluted HF (e.g., Li & Li, 2011; Buylaert et al., 2013). However previous studies showed that etching does not remove a uniform layer around feldspar grains but rather the acid penetrates along cleavage planes (Duller, 1992, p. 16). K-content in KF is either measured (Porat et al., 2004) or assigned a fixed value, usually 12.5 % (Huntley & Baril, 1997). As we will show, etching with HF might have bearings also on the K-contents of the KF.

Extraction procedures are often described briefly, even in the more applied, hand-on reviews and books (e.g., Wintle 1997, Aitken 1998, p. 66–67), and many of these protocols may have not been tested in years, if ever. This study brings together the results of a series of experiments that test various steps in conventional preparation protocols used to extract quartz and KF. The topics investigated were:

1. Is magnetic separation as effective as the 2-step density separation for producing pure quartz extracts?
2. Does conventional HF etching (40–48 % HF for 40 min) remove the 10 μm zone affected by α-particles?
3. How common are precipitated fluorides and do we need the ensuing step of dissolving them in HCl?
4. What are the effects of etching with diluted HF on feldspars?
   (a) By how much is grain size reduced?
   (b) Does etching uniformly remove the α-affected zone?
   (c) Does the D_e change with etching?

One important property of clastic sediments commonly dated by luminescence methods is their maturity, which is reflected among other things in the presence of rock fragments and the quantity and type of feldspars and heavy minerals. Sediments mature physically by repeated deposition/resuspension and long distance transport of the sediment grains from their source; this results in a reduction in grain size and the preferential breakdown and removal of unstable minerals and rock fragments (Blatt & Tracy, 2005). For quartz it entails the disintegration of grains rich in other mineral inclusions and air bubbles, leaving behind the more resistant grains. For feldspars the removal of the mineralogically less stable grains (e.g., plagioclase) and altered (cericitised) grains results in a K-rich and resilient feldspar fraction. This process appears to increase some desirable luminescence properties of quartz, such as brightness and sensitivity (Pietsch et al., 2008). In this study we tested sediments of high and low level of maturity, to see how each responds to conventional laboratory procedures.

2. Samples and laboratory methods

Coarse grain quartz and KF were extracted from three samples from the collection of the Geological Survey of Israel luminescence dating laboratory, of different geological setting and mineralogical maturity:

- RAM-8: mature, fine sandy, quartz-rich sediment derived from reworked dunes in the western Negev, Israel, with the sand originating from the Nile (Ben-David, 2003). K-rich feldspars were concentrated from this sample.
- FGA-26: coarse desert loess from the Negev Mts. (Faerstein, 2003), for which the source of the quartz is distant dunes (Enzel et al., 2010) with mature mineralogy (Muhs et al., 2013). Quartz was extracted from this sample.
- 23-5: Fluvial sand from Nahal Avrona, southern Negev (Amit et al., 2002), derived from a nearby igneous terrain, containing abundant rock fragments and heavy minerals. Both quartz and K-rich samples were separated from this mineralogically less mature sample.

After sieving each sample to the desired grain size and dissolving carbonates with 8 % HCl, the ensuing treatments varied. The different treatments and analyses performed on each sample are listed in Table 1 and the captions therein.

Scanning Electron Microscope (SEM) imaging was used to observe the effects of etching on the surface morphology of quartz and KF grains. Energy Dispersive Spectrometer (EDS) was used to check the purity of the quartz extracts by mapping K and Na contents, and to identify fluorides and analyze their composition in HF-etched quartz and KF. Grain size analysis before and after HF-etching, measured using a laser Malvern Mastersizer, was used to check the reduction in grains size due to etching. An XRF module attached to a Riso reader was used to measure the relative K-contents (of the sum K+Na+Ca) in the different separated and etched KF fraction (Kook, 2012). D_e was measured on large (8 mm) multi-grain aliquots using the SAR protocol for quartz (Murray & Wintle, 2000) and the post IR-IR$_{290}$ SAR for KF (Thiel et al., 2011).

Preparation						Analyses			
Sample	Grain size (μm)	Density separation		Magnetic separation	HF [Strength (%) / duration (min)]	HCl 16%	D_e	SEM	XRF	Grain size
		2.62 (g/cm^3)	2.58 (g/cm^3)							
Alkali Feldspar										
RAM-8	149–177	x	x		0		x	x	x	
		x	x		10/10		x	x	x	
		x	x		10/20		x		x	
		x	x		10/40		x	x	x	
23-5	125–177	x	x		0		x	x	x	x
		x	x		10/10		x	x	x	
		x	x		10/20		x		x	x
		x	x		10/40		x	x	x	
Quartz										
23-5	125–177	x			0			x		x
		x			40/40			x		
		x			40/40	x	x	x		x
23-5	177–212			x	0			x		x
				x	40/40			x		
				x	40/40	x	x	x		x
FGA-26	74–125			x	0			x		x
				x	40/40			x		
				x	40/40	x	x	x		x
				x	40/60					x

Table 1. Samples used and analyses carried out in this study. The samples were sieved to the required grain size, soaked in 10 % HCl overnight to dissolve carbonates, rinsed and dried. This was followed by either density separation (Sodium-Polytungstate with density of 2.62 g/cm^3) or magnetic separation (Frantz LB-1, 1.4 A on the magnet). HF etching was carried out for the durations and concentrations listed, which for some samples was followed by rinsing in 16 % HCl overnight. All preparations and analyses were performed at the laboratories of the Geological Survey of Israel, except for XRF that was measured in Risø-DTU using a modified Risø Reader with an XRF attachment (Kook, 2012). Grains size was measured with Malvern Mastersizer laser particle size analyzer.

3. Results

SEM/EDS mapping showed that before HF-etching, all quartz samples contained small amounts of feldspars (3–10 %), indicating that neither density separation nor magnetic separation can fully remove all feldspars, and that HF etching is essential. SEM/EDS mapping also showed that the alkali feldspar extracts contained some quartz grains (∼5 %). Roughly15 % Na-rich grains were observed within the predominantly K-rich feldspars of sample 23-5.

3.1. Comparison of extraction procedures for quartz

For sample 23-5, SEM images followed by EDS mapping of Si and K, showed that the two size fractions (125–177 μm and 177–212 μm), each extracted by a different procedure (and followed by HF-etching), contained pure quartz, with no contaminating KF (Fig. 1). The measured D_e values (on 24 aliquots for each size fraction) were statistically identical, 10.8 $\pm$ 1.0 Gy and 9.7 $\pm$ 1.6 Gy for the 125–155 μm and 177–212 μm, respectively.

3.2. Reduction in grain size

Grain size reduction of quartz due to etching in 40 % HF for 40 min varies among samples (Fig. 2). The mineralogically mature sample FGA-26 was etched by ∼10 μm; increasing etching time to 60 min did not reduce grains size any further. As the shape of the particle size distribution did not change much by etching, only the peak shifted by 10 μm (Fig. 2a), we can deduce that reduction in grain size was fairly uniform. In the two grain size fractions of the less mature sample 23-5 (125–177 μm and 177–212 μm), 40 min etching reduced the modal grain size by 33 μm and 48 μm, respectively. Etching also resulted in broadening of the particle size distribution and an increased tail of smaller particles

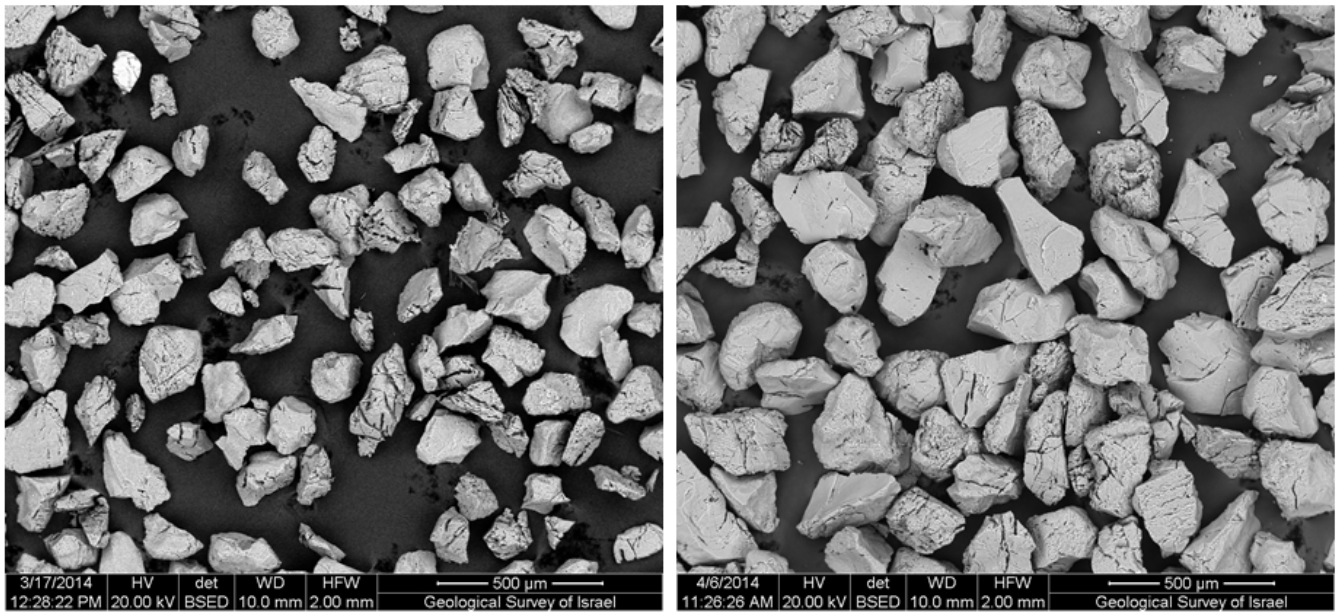

Figure 1. SEM images of two size-fractions of quartz sample 23-5, purified by different procedures. a. 125–177 μm, extracted using 2-step density separation (2.62 g/cm^3 and 2.58 g/cm^3), followed by HF etching and rinsing with HCl; D_e = 10.8 $\pm$ 1.0 Gy. b. 177–212 μm, extracted using magnetic separation, followed by HF etching and rinsing with HCl; D_e = 9.7 $\pm$ 1.6 Gy. Average D_e values are statistically identical, however the coarser grain size has higher scatter on the average D_e.

(Fig. 2b).

Etching KF sample 23-5 (125–212 μm) in 10 % HF for 20 min and 40 min resulted in grain fragmentation (Fig. 3c), and the modal grain size was reduced by 50 μm and 100 μm, respectively (Fig. 2c). Particle size distribution after etching is substantially shifted towards the smaller grains when compared to the original distribution, indicating that most grains had their size reduced, though perhaps not uniformly.

3.3. Removal of feldspars in quartz by HF

Soaking quartz in 40 % HF for 40 min was very efficient in removing all feldspars, regardless how much was previously present. SEM images and EDS mapping show no remaining KF (Fig. 1).

3.4. Fluoride precipitation and removal

In both mineral extracts, HF etching resulted in fluoride precipitation, observed using the SEM/EDS. These are shown in Fig. 3; the chemical composition of the precipitates is given in Table 2; and their EDS spectra are shown on Fig. 4. In the quartz extracts, poorly crystalline Ca-fluorides precipitated (Fig. 3e & 4c). Subsequent soaking of the HF-etched quartz samples in 16 % HCl overnight completely removed all Ca-fluorides (Figs. 1 & 3f).

In KF sample 23-5, well crystallized, idiomorphic K-fluoride crystals formed after 40 min etching in 10 % HF (Figs. 3c & 4b). In sample RAM-8 fluorides appeared as powdery coating of the KF grains (Fig. 3d). Subsequent soaking in HCl was not checked for the removal of K-fluorides precipitated in the KF samples.

(a)			
Element	**Weight %**	**Atomic %**	**Number of ions**
F	61.49	66.65	6.24
Na	0.61	0.55	0.05
Al	8.69	6.63	0.62
Si	1.62	1.19	0.11
Ca	11.68	6.00	0.56
Ba	1.31	0.20	0.02
O	14.60	18.79	1.76
Totals	100.00		
(b)			
Element	**Weight %**	**Atomic %**	**Number of ions**
F	29.82	38.06	4.62
Si	8.26	7.13	0.87
K	43.60	27.04	3.29
O	18.33	27.78	3.38
Totals	100.00		

Table 2. SEM/EDS analyses of selected precipitated fluoride crystals. (a) Ca-fluorides in HF-etched quartz sample FGA-26 (Fig. 4e); (b). K-fluorides in HF-etched KF sample 23-5 (Fig. 4c).

3.5. Uniformity of etching

SEM images show the manner in which HF dissolved the quartz and KF grain surfaces. In quartz, the mature sample

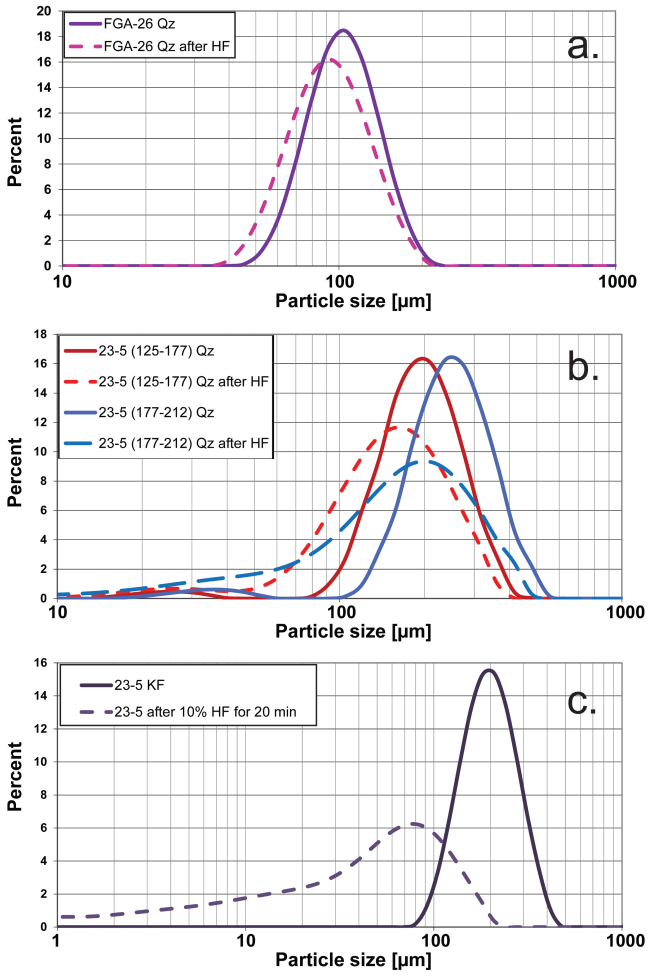

Figure 2. Grain size analyses (average of 3 measurements) before (solid line) and after (dashed line) etching. Quartz (a. and b.) was etched with 40 % HF for 40 min. a. 88–125 μm quartz extract, sample FGA-26. Etching reduced the modal grain size by 10 μm. b. 125–177 μm (red) and 177-212 μm (blue) quartz extracts, sample 23-5. Modal grain size was reduced by 33 μm and 48 μm, respectively. Note broadening of particle size distribution and increase in the proportions of smaller particles after etching. c. 125–212 μm KF extract, etched with 10 % HF for 20 min. Etching resulted in grain fragmentation and the modal grain size was reduced by 50 μm.

FGF-26 was etched in a more even manner when compared to the less mature sample 23-5 (compare Figs. 3e and 1b). In the latter, some quartz grains were etched preferentially along fissures and grain boundaries (Fig. 1), possibly because these grains contain mineral inclusions and air bubbles which are more susceptible to etching.

For the KF, etching was preferential along cleavage plains, becoming more severe with longer etching time. Ten min etching of the mature sample (RAM-8) had only a minor effect on the morphology of the grains, whereas that same duration of etching severely affected sample 23-5. Forty min etching of the latter resulted in severe grain fragmentation (Fig. 3c).

3.6. Effects of etching on the D_e values of KF

Removing the outer zone of grains should reduce their measured D_e values as the area dosed by α-particles is removed. For both KF samples (23-5 and RAM-8) the D_e values, measured using the post IR-IR$_{290}$, were reduced as a function of etching time (Figs. 5a & 5b) and after 40 min they were 10–15 % lower than that of the unetched samples. The reduction expected due to removal of 20 μm was calculated from the dose rates for the individual samples, and is shown on Fig. 5 as a horizontal bar. For sample 23-5, 10 min etching was sufficient to reduce the D_e value as expected; further etching has only a minor affect. However the D_e value for sample RAM-8 continues to decrease with longer etching time, to values well below those expected from the removal of the α-affected zone. This could be explained by the removal of a weathering rind or a thin clay mineral coating, not identified by the SEM or XRF.

3.7. Effects of etching on K-contents in KF

K-content was measured for the original, unetched KF extracts using inductively coupled plasma spectroscopy (ICP-AES), and the relative K-contents (as a fraction of the total K+Na+Ca) were measured for the original and etched fractions using XRF. For sample 23-5, etching increased relative K contents in the KF fraction by ~30 %, from 0.70 to 0.93 (a value of unity signifies pure orthoclase with 14 % K) and lowered the Na contents (Fig. 5c), which can be explained by preferential dissolution by HF of the Na-feldspar remaining in the sample after density separation. The increase in the K-contents by ~30 % (from the original value of 7.2 % as measured by ICP-AES) brings this value closer to the "ideal" 12.5 % commonly used for internal dose rate calculations (Huntley & Baril, 1997). On the other hand, for sample RAM-8, with original K-contents of 11.5 %, 40 min etching somewhat lowered the relative K-contents from 0.94 to 0.88, which can be explained by the removal of K-rich clay coating (also deduced from the reduction in D_e by etching).

4. Summary and Recommendations

In this study we found that routine HF etching of quartz (40 % HF, 40 min, ~5cc HF for each gram of quartz) removed 10 μm from geologically mature samples; increasing etching time to 60 min did not reduce grain size any further. The modal grain size of mineralogically less mature quartz samples was reduced with etching by 30–50 μm, possibly due to fragility of quartz grains. Although small, the α-dose rate remaining after the removal of 10 μm is not zero and it should be taken into account when calculating ages for some quartz samples, particularly those with low dose rates.

Ca-fluorides precipitated during HF etching in all quartz samples but were fully removed by soaking overnight in 16 % HCl. More experiments are needed to see if higher concentration of HCl coupled with shorted immersion time can achieve the same result. Also the effect of these precipitated fluorides on the luminescence signal and the measured

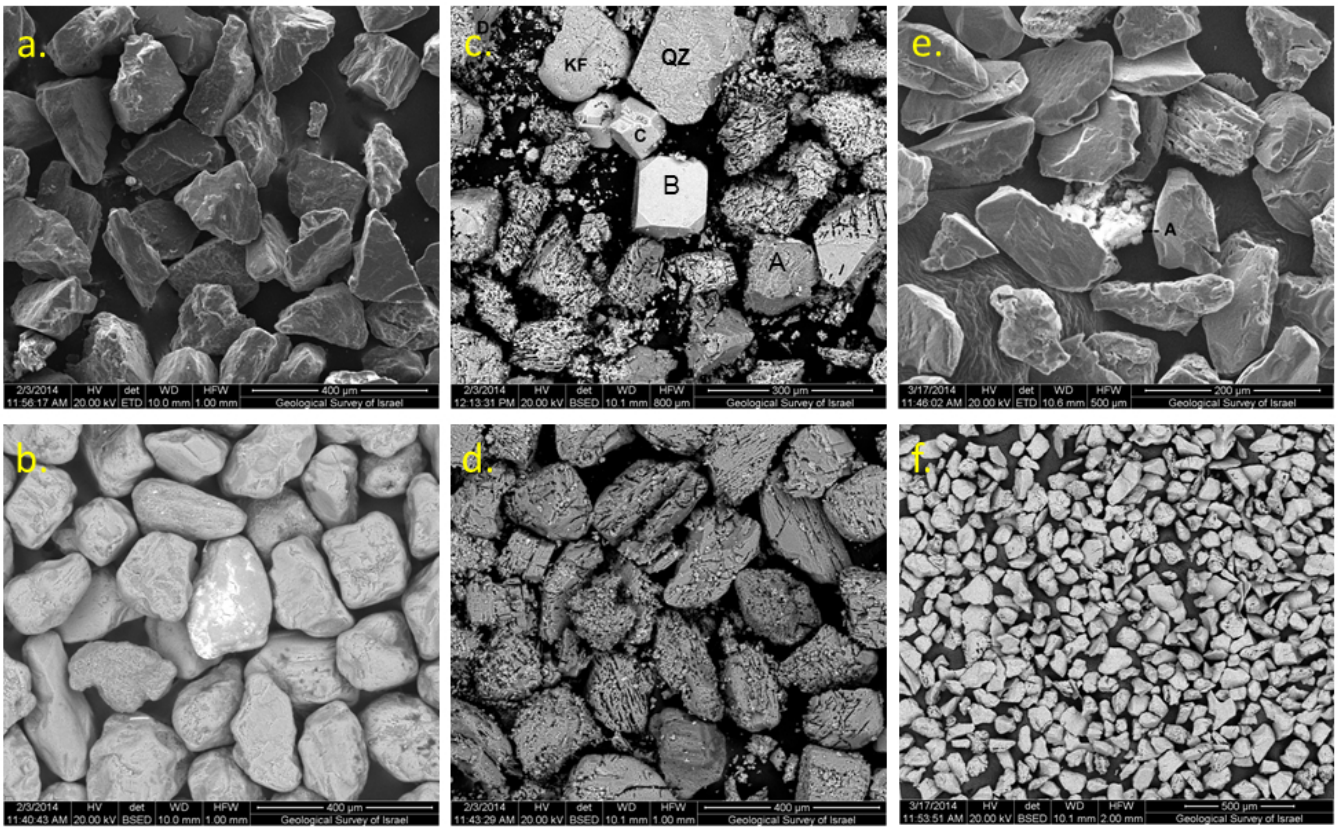

Figure 3. SEM/EDS images showing the effects of HF etching on KF and quartz. Unetched KF, 23-5 (a) and RAM-8 (b). Note angular and sharp grains in the less mature sample 23-5 against rounded and smooth grains in the mature RAM-8 samples. The same samples after 40 min etching in 10 % HF are shown on c. (23-5) and d. (RAM-8). For 23-5 (c), note highly pitted and disintegrated KF grains, isolated unaffected quartz (marked QZ) and KF (marked KF) grains, and newly precipitated idiomorphic, cubic fluoride crystals (marked A, B, C). Representative EDS spectra of the KF grain and crystal B are given in Fig. 5. Chemical composition of grain B is given in Table 2b. For RAM-8 (d), the grains are intact but are deeply etched along cleavage plains. The fluorides appear as fine crystals on the KF grain surfaces. e. and f. Quartz sample FGA-26 after HF etching, before (e) and after (f) rinsing overnight in 16 % HCl. In e. fluorides appear as white powder in the center. Representative EDS spectra and analyses are given in Fig. 5c. and Table 2a. After etching and soaking in HCl (f) no fluorides were observed.

D_e values need testing.

Etching KF, even with diluted (10 %) HF, affected the mineral grains in a complex manner. It resulted in uneven reduction in grains size so that the α-affected zone might not be removed in a fully uniform manner. Extended etching also created severe micro-scale corrosion along cleavage plains, grain disintegration and substantial precipitation of K-fluorides. The reduction of 100 μm in the modal grains size after 40 min etching is probably due to grain breakage along etched plains.

Nonetheless, particle size distribution show that the modal grain size is substantially reduces (50 μm after 20 min), suggesting that even though some grains are etched more than others, we can assume that for most grains at least 10 μm were removed. Additionally, etching cleans unwanted coatings from the grains and lowers D_e values sufficiently to assume that the α-affected zone is removed; and in cases it increased average K contents, probably by preferentially dissolving Na-feldspars. Thus, mild etching of KF is beneficial in several aspects.

Overall, the results presented here provide confidence in standard laboratory sample preparation procedures. More samples from other geological provinces should be tested in the manner presented here to fully confirm the presented observations.

Here are suggested modifications of the standard procedures for optimal quartz and KF purification:

- More concentrated HF might be considered if grains are to have their entire α-affected area (20 μm) removed. Otherwise one should not assume that the entire zone was etched.
- Soaking the HF-etched quartz grains in HCl (in this study it was overnight in 16 % HCl) is necessary to remove all fluorides and avoid the risk of these contributing to the luminescence signal of quartz.
- Since HF removes all feldspars in quartz-rich samples, there is no need to re-sieve the samples after HF, as that would bias the original grain size and the values

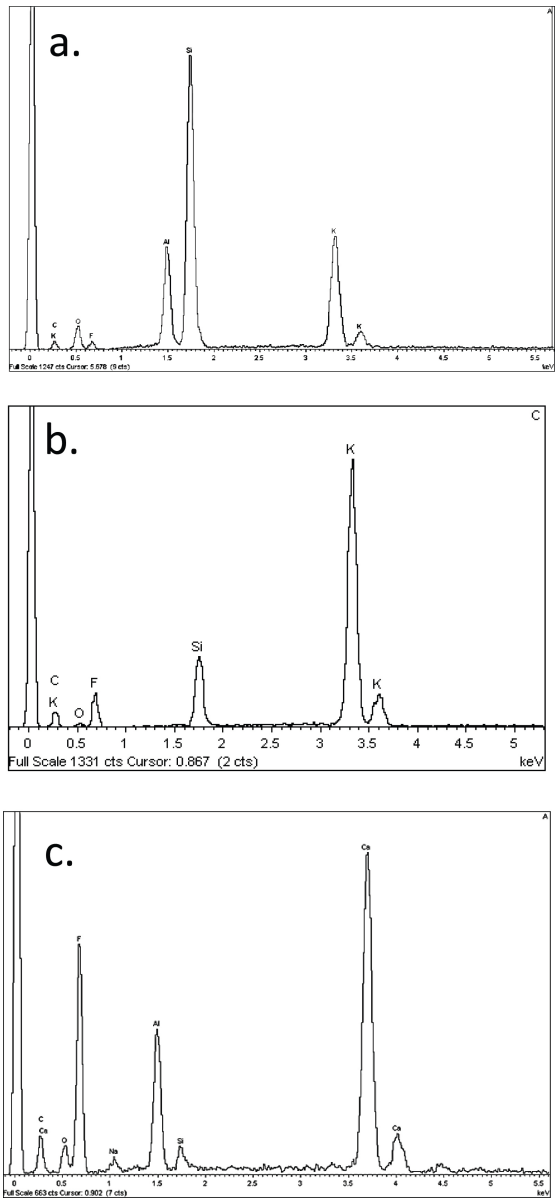

Figure 4. SEM/EDS composition spectra of selected mineral grains. a. Typical K-feldspar, sample 23-5. b. K-fluoride precipitate (see also Table 2b), sample 23-5. c. Ca-fluoride precipitate (see also Table 2a), sample FGA-26.

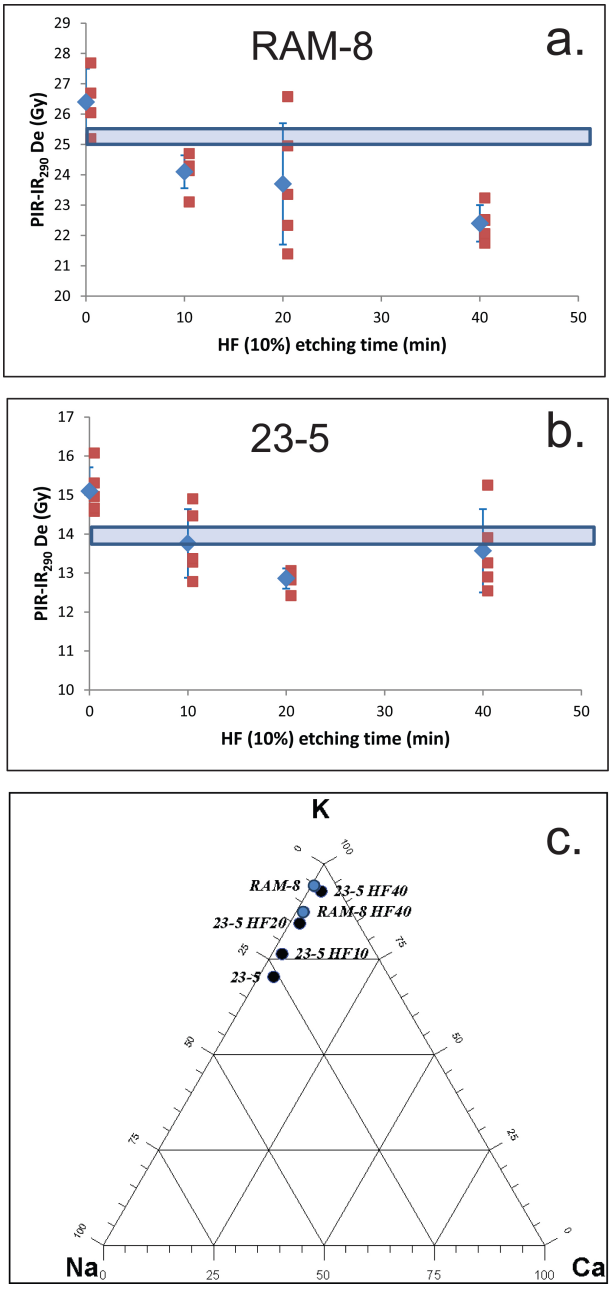

Figure 5. D_e values and chemical composition measured on KF extracted from two samples (using 2-step density separation), etched with 10 % HF for varying durations. a. & b. D_e values as a function of etching time for KF extracts of RAM-8 & 23-5, respectively. The horizontal bar shows the D_e expected after removing the 20 μm zone affected by α-particles, calculated from the original D_e (0 time etching), the sediment dose rate, 12.5 $\pm$ 0.5 % K in the KF and an a-value of 0.1 $\pm$ 0.05. Note that for sample RAM-8 (a) the reduction in D_e is greater than that explained by etching 20 μm. c. Ternary diagram of XRF measurements of KF extracts showing changes in relative K, Na and Ca contents (representing the feldspar minerals Orthoclase, Albite and Anorthite, respectively). For sample 23-5 (dark blue circles) etching substantially increased the concentration of K while reducing Na contents. For sample RAM-8 (light blue circles) 40 min etching somewhat reduced K contents.

used for calculating beta attenuation. But if sieving is done, a sieve size smaller by at least one mesh than the lower grain size should be used, so that only fragmented grains are removed.

- Caution should be used when etching KF; it is recommended to use diluted (no more than 10 %) HF for a short duration (10–15 min). This short duration will minimize K-fluoride precipitation and will most likely remove most of the α-affected zone. Longer etching might compromise some of the KF properties.

- An added step of soaking the HF-etched KF in 16 % HCl overnight to remove fluorides is advisable.

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Reviewer's Comment

1. This study demonstrates that sediment maturity, a concept developed in sedimentary petrology, has a definite link to mineral solubility in HF and hence, would explain some of the variability seen in the purity of HF-treated quartz extracts. 2. Routine luminescence dating laboratory procedures should include laser diffraction particle size analysis as it allows for more precise assessments of both equivalent dose and dose-rate.