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HF treatment for the isolation of fine grain quartz for luminescence dating

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Abstract: *Optical dating of silt sized quartz is advantageous for fluvially transported sediments where the extent of pre-depositional zeroing is uncertain. The effect of sunlight on the OSL of quartz is faster, than for feldspar, and the silt sized particles being carried closer to the surface of water than sand sized ones will have received a greater sunlight exposure. An HF treatment for isolation of quartz, requiring only a few hours, is described. This method has been found to be useful for samples containing up to 40% feldspar.*

Introduction

The extent of zeroing of a luminescence signal is uncertain in the case of fluvially transported sediments due to the attenuation of the solar flux due to water depth (Berger and Luternauer, 1987; Berger, 1990), sediment load (Jerlov, 1976; Berger, 1990; Ditlefson, 1992), turbulence (Gemmel, 1985) and duration of transport. In addition different grains are likely to have had different histories of light exposure on account of their different sizes, source regions, and length, duration, and mode of transport and deposition. Silt-sized grains are carried closer to the top of the water surface on average, and settle more slowly than sand-sized grains; they thus have a greater probability, not only of longer duration exposure to photons, but also to a wider spectrum and hence being well bleached. In cases where the extent of pre-depositional bleaching is uncertain it makes sense to use the fine grained quartz for optical dating as comparative studies of bleaching by sunlight for OSL and TL by Godfrey-Smith et al (1988) have shown that for OSL, 1% of the initial signal was reached in a few seconds for quartz and a few minutes for a sample of feldspar. The feasibility of OSL dating of fine grained quartz from sediments has been demonstrated by Rees-Jones (1995) who used fluorosilicic acid, following the procedure given by Jackson et al (1976), to eliminate feldspars. This method however takes several days for completion. Here, an HF treatment procedure for dissolution of fine grained feldspar from polyminerallic samples within a few hours is described.

Materials and Methods

Museum samples of quartz and orthoclase feldspar were taken and powdered. The powdered samples were subjected to routine treatments as for natural samples. They were sequentially treated with HCl, and hydrogen peroxide, and deflocculated using a sodium oxalate solution. The 4-11μm size fraction was then isolated using Stokes' settling in acetone. The samples were tested for purity using X-ray diffraction. Quartz was additionally tested for purity using infra-red stimulation and yielded count rates close to background. The 4-11μm quartz and orthoclase samples were dried and weighed. They were then made up to equal concentration suspensions and mixed in differing proportions to obtain mixtures with 20% feldspar (20%F), 40% feldspar (40%F) and 80% feldspar (80%F). These were subjected to the treatments outlined in Table 1.

IRSL was measured using a Risφ system, using the filters BG39, 7-59 and HA3, which show a maximum transmission around 365 nm, the emission band of quartz (Huntley et al, 1991). A pre-heat treatment of 220°C for 1 min was used.

Table 1: *Treatment for 4-11 μm mixture samples.*

Sample	Treatment*	ß Dose (Gy)	Total photon counts§ in first 10 seconds
20% F	Unetched. Deposition on Al discs. Sunlight exposure for 3 hrs.	90	12720
		300	38741
	5% HF etch for 80 min followed by HCl wash for 120 min. Removal of <4μm using Stokes settling. Deposition on Al discs. Sunlight exposure for 3 hrs.	90	502
		300	604
	5% HF etch for 120 min followed by HCl wash for 120 min. Removal of <4μm using Stokes settling. Deposition on Al discs. Sunlight exposure for 3 hrs.	90	615
		300	716
40% F**	Unetched. Deposition on Al discs. Sunlight exposure for 3 hrs.	90	24635
	10% HF for 120 min followed by HCl wash for 120 min. Removal of <4μm using Stokes settling. Deposition on Al discs. Sunlight exposure for 3 hrs.	90	510

*All etch treatments were carried out in 4 cm liquid column to enable feldspar dissolution and allow quartz grains to settle.

§ The total photon count in first 10 seconds for discs subjected to 3 hours sun exposure only were around 537 counts.

** Preliminary studies indicated that while 5% HF was unable to remove all feldspar from mixtures containing more than 20% feldspar, treatment with stronger than 10% HF acid resulted in complete dissolution of sample, including the quartz. Therefore, only 10% HF was used for samples with more feldspar.

Results

As shown, Fig.1 indicates the etched samples do not show any IRSL signal even after being irradiated to high doses (90 or 300 Gy) indicating the absence of feldspars. The unetched 20% feldspar mixture shows significant photon counts for the same beta dose. Since these aliquots were made from separate batches of samples, normalisation was not attempted; rather an absence of IRSL signal was considered as a measure of efficiency of the treatment in removing orthoclase feldspar. Similar results were obtained for 40% feldspar mixtures. Initial tests with 80% feldspar mixtures were unsuccessful and high photon counts were observed even after etching for 120 min using 10% HF. Since both 80 min and 120 min HF treatment succeeded in removing the feldspars, an 80 min treatment for samples having low feldspar amount (~20% feldspars) is recommended. Longer treatment may be attempted for samples with higher feldspar amount but all the quartz may be dissolved. Experience thus suggests that only samples with up to 40% feldspar have a high chance of success using this technique.

The drawback of this technique is that some amount of quartz is also lost and larger sample amounts may be required for dating. This method has been successfully used to remove feldspar from fluvial sediments from Nal region, in Gujarat State, NW India. However, it is recommended that natural samples be routinely tested after the HF treatment using IR stimulation.

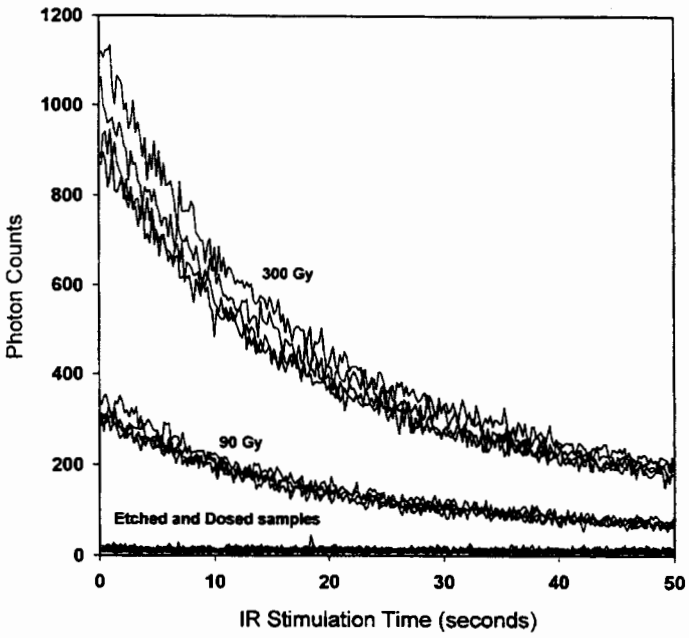

Figure 1.
Plot showing the photon counts in response to IR stimulation for samples containing 20% feldspar, before and after etching. Dwell time was 0.2 seconds.

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Rewiever

D.J. Huntley

Comments

This appears to be a useful and simpler alternative to the H2SiF treatment. It would be nice to know how much of the quartz is lost. I am puzzled as to why the proportion of feldspar grain is surrounded by acid. Perhaps one needs to ensure an adequate amount of acid and adequate stirring.

Finally there is the question of zircon. It will not be removed by the acid treatment and will be measured along with the quartz. We know that small amounts of zircon can dominate "quartz" thermoluminescence if the zircon is not removed first. What is the situation with optical excitation?