

— Report —

Experiments on method for washing drill cuttings: evaluation of soaking, stirring, and milling effects

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Effects of soaking, stirring, and milling were investigated to standardize the washing method used for drill cutting samples. Drill cuttings are fragments of solid material ground by a drill bit at the bottom of a hole. They are recovered on the ship in riser drilling as a component of the circulating drilling mud. Removal of drilling mud from the surface of cutting samples must be done before cutting samples are analyzed. Cutting samples from IODP Expedition 337 were used in test experiments. The samples were analyzed before and after soaking, stirring, and milling with XRD, XRF, an elemental analyzer and a carbonate analyzer. Results show that Na and K contents are reduced by the removal of drilling mud. However, changes that occur after 6 h of soaking are rather minor. These results suggest that stirring of cuttings in heated seawater at 40°C for 6 h after quick washing is effective for XRF and TOC analyses.

Keywords : drill cuttings, ocean drilling, contamination, drilling mud

Received 15 January 2015 ; Revised 3 December 2015 ; Accepted 14 December 2015

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1. Introduction

Drill cuttings are fragments of solid material that are produced when a drill bit penetrates into a formation at the bottom of a hole. In riser drilling, cuttings are removed from the borehole, after which they are transported to the surface together with drilling mud. Cutting samples are useful for lithology description, and are particularly important when core samples are unavailable or only partially available.

Cuttings have been utilized in previous deep drilling projects, mainly for lithology identification and studies on rock physical properties. In the German Continental Deep Drilling Program (KTB), a suite of petrophysical properties were obtained from cuttings (see review by Berckhemer et al., 1997; Emmermann and Lauterjung, 1997). In the San Andreas Fault Observatory at Depth (SAFOD), intensive analyses were conducted on cuttings sample from fracture and fault zone (e.g., Carpenter et al., 2011; Morrow et al., 2007). In addition to physical properties, mineralogical and geochemical analyses provided insights into in-situ faulting process and fluid-rock interactions (Schleicher et al., 2006; 2009). These studies show the value of cuttings from a deep hole where core samples are available only sparsely.

In scientific ocean drilling, the scientific use of cutting samples started when Chikyu began riser drilling in 2009. Some of the pioneer studies were carried out successfully using cuttings samples from Nankai Trough Seismogenic Zone Experiment (NanTroSEIZE) expeditions (Noguchi and Okamura, 2014; Takeshita et al., 2014). On the other hand, it was pointed out that the data from core samples and cuttings samples show differences; e.g., inorganic carbon content of cuttings was higher than those

of core samples of IODP Expedition 337 (Expedition 337 Scientists, 2013a). The cause of the difference was, however, not fully discussed and standard protocol for the scientific use of cutting samples is not yet fully developed.

One difficulty of using cuttings samples for scientific analyses is that cuttings are more prone to contamination by drilling mud than core samples are. Small cuttings are fully coated by drilling mud during their transport from the borehole to the surface. It is not easy to remove the drilling mud from the cuttings, even after repeated washing. Removal of drilling mud is a crucially important problem when using cuttings for chemical analyses.

This report describes our attempt at different methods of washing cuttings, with a view to improve the routine protocol for washing a large amount of cuttings samples. Three treatments were attempted: (1) soak cuttings in seawater, (2) soak and stir cuttings in heated seawater, and (3) mill cuttings and soak the powdered cuttings in seawater. To evaluate the treatment methods, we examined the variations of chemical compositions of the washed cuttings.

2. Cutting, seawater, and drilling mud

Two cutting samples taken from Hole C0020A during Expedition 337 were used for this study. Sample 352SMW was taken from 2296.5–2306.5 m mud depth below the seafloor (mMSF). Sample 385SMW was taken from 2436.5–2446.5 mMSF (Table 1). Both intervals correspond to Unit IV in the hole (Expedition 337 scientists, 2013a). After the expedition, each sample was stored

Table 1. List of cutting samples and processing methods used for this study. RT, room temperature

Sample	Source	Lithology	Size fraction	Processing			
				Initial wash	mill	Soak in RT	Stir+warm
A-1	352SMW 2296.5- 2306.5 mMSF	Silty shale	1-4 mm	yes	-	-	-
A-2					-	6 h	-
A-3					yes	-	-
A-4					-	-	6 h
B-1			-		-	-	
B-2			-		24 h	-	
B-3			yes		-	-	
B-4			-		-	24 h	
C-1	385SMW 2436.5- 2446.5 mMSF	Silty shale + Sandstone	> 4 mm	yes	-	-	-
C-2					-	3 h	-
C-3					yes	-	-
C-4					-	-	3 h

unwashed in 1 L-polyethylene container. Each was then kept at 4°C in a refrigerator for about 8 months.

Seawater used for this study was taken on May 22, 2013 when the *Chikyu* was located off Sado Island in the Sea of Japan. The seawater was put into a 20 L polyethylene container and was kept at room temperature in the Chikyu laboratory, which is air-conditioned at 20°C.

Riser drilling mud used in Exp. 337 was composed of KCl, NaCl, bentonite, barite (BaSO₄), polymer derived from cellulose, starch and polypropylene glycol, and sulfonated asphalt as hole stabilizer. Other minor components such as remains of lost circulation materials include mica, calcium carbonate, and grains of organic origin. These were mixed to prepare drilling mud with density maintained at 1.06–1.11 sg. The components and their concentrations were variable through drilling at Hole C0020A. In addition to reports from a mud engineer, several drilling mud samples were analyzed during Exp. 337. Table 2 shows results obtained for 373LMW, the most relevant drilling mud sample, taken between the sampling of 352SMW and 385SMW.

Experiments described in the following section were conducted during May–June, 2013 in the Chikyu laboratory.

3. Sample preparation and instruments

The sample preparation flow is presented in Fig. 1. A batch of 120 cc of cutting samples was quickly washed at once in seawater and was sieved with 0.25 mm mesh. The samples of >0.25 mm were sieved again in seawater for separation of 1–4 mm and >4 mm size fractions. The second sieving duration was 30 s. Iron grains, which were regarded as broken pieces of drill bit and drilling equipment, were removed using a hand magnet. Next, the sample batches were washed and sieved until the required volume of samples was obtained. To minimize variation in lithology, fragments of similar lithology were picked from the >4 mm size fraction. When the collected volume was insufficient, the 1–4 mm fraction was also used. In all, 160 cc of dark grayish-

brown silty shale was selected from 352SMW to prepare eight 20 cc samples: Samples A1–A4 and B1–B4 in Table 1. From 385SMW, 40 cc of grayish brown to dark grayish brown silty shale, and another 40 cc of bluish gray to dark bluish gray sandstone were selected. Those were divided into four 10 cc samples respectively and were mixed to prepare four 20 cc samples: Samples C1–C4 in Table 1.

Each sample set A–C was divided into four subsamples. Subsample 1 was vacuum-dried immediately without further washing. Subsample 2 was soaked in 200 cc of seawater for fixed periods; then they were vacuum dried. Subsample 3 was soaked in 200 cc of warm (approx. 40°C) seawater with a stirring bar, and were stirred gently for the same period as Subsample 2. Subsample 4 was milled in an agate mortar. Then the milled powder was soaked in 200 cc of seawater for the same period as that used for Subsample 2.

The soaking time was 6 h for sample series A, 24 h for sample series B, and 3 h for sample series C. After

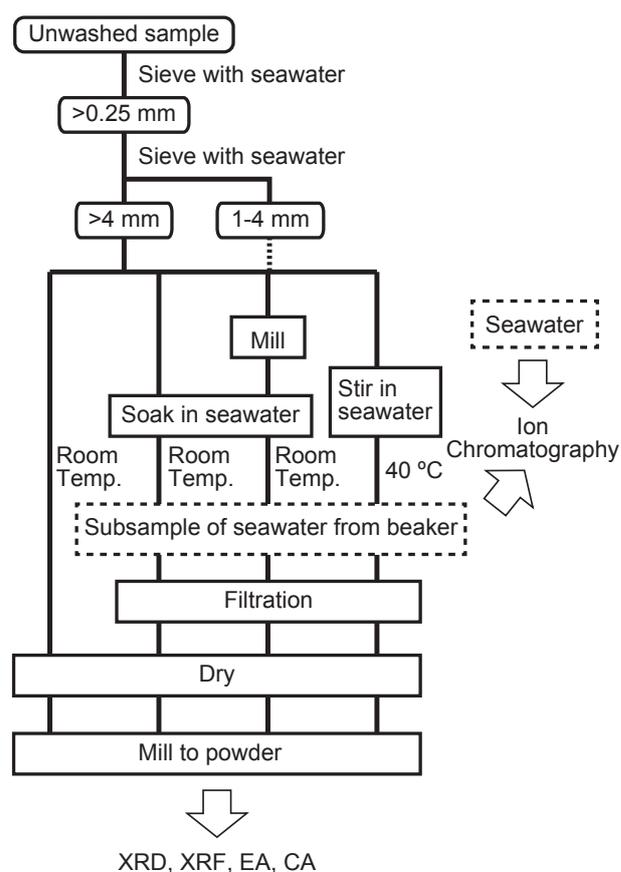


Fig. 1. Four washing methods were used for this study: washing during sieving, soaking in seawater, soaking milled sample in seawater, and stirring in seawater. All samples were dried and milled for measurements. EA, elemental analyzer; CA, carbonate analyzer.

Table 2. Analysis of drilling mud sample 373-LMW (from Table T12, Expedition 337 Scientists, 2013a).

sample	Na ⁺ (mM)	K ⁺ (mM)	Mg ²⁺ (mM)	Ca ²⁺ (mM)	Cl ⁻ (mM)	SO ₄ ²⁻ (mM)
373-LMW	1019.8	668.84	2.23	4.05	1394.39	50.99

soaking, samples were filtered and vacuum-dried for 24 h. Then they were milled using a ball mill at 200 rpm for 6 min.

After the cuttings were removed, 2 ml of the soaking seawater was sampled to examine changes in the chemical composition. The subsampled seawater was divided into two portions. Then 6 M HCl was added to one for cation analysis.

The cutting samples were measured using XRF, XRD, an elemental analyzer (EA), and a carbonate analyzer (CA) to ascertain the major element concentrations, mineral composition, total carbon, organic carbon, nitrogen and sulfur contents. We also used ion chromatography to ascertain the concentrations of cation and anion in seawater samples. The measurement instruments and protocol, unless otherwise noted, are the same with those used in Exp. 337 (Expedition 337 Scientists, 2013b).

4. Results

4.1 Major elements

Major element concentrations from XRF data are presented in Table 3. The concentrations of the 10 major elements (Na_2O , MgO , Al_2O_3 , SiO_2 , P_2O_5 , K_2O , CaO , TiO_2 , MnO , and Fe_2O_3) are corrected by multiplying $(100-\text{LOI})/100$ to include volatile components, where LOI is the loss on ignition. Results show that Sample C, containing sandstone, has higher concentrations of SiO_2 and Na_2O than either Sample A or Sample B.

The soaking effect is revealed by comparison between subsamples 1 and 2. The concentrations of Na_2O ,

K_2O and LOI decrease after soaking in all three samples. Stirring in warm water enhances the decrease in K_2O concentration, but does not strongly affect Na_2O and LOI. The effects of milling are characterized by the increase in Na_2O concentrations. MgO and CaO increased in all samples regardless of the washing methods. Changes in other elements are minor and are relative variation due to the major changes described above.

4.2 Total C, N, S, and TOC

Table 4 presents the contents of total carbon (TC), total nitrogen (TN), total sulfur (TS), inorganic carbon (IC), and total organic carbon (TOC). TOC is calculated by subtracting the IC from the TC. With the exception of TS, all contents are higher in Samples A and B than in Sample C.

The TC and TOC contents decrease 0.6–0.8% during soaking with seawater. The TS contents also decrease by soaking, although changes in TN and IC contents are not significant. The milling effects show a further decrease in TC and TOC, but the effects of stirring are inconsistent.

4.3 Mineral composition

Table 5 presents X-ray counts for representative minerals. All samples were composed mainly of quartz ($2\theta = 26.7^\circ$), albite ($2\theta = 28.0^\circ$), orthoclase ($2\theta = 31.8^\circ$), chlorite-serpentine ($2\theta = 12.5^\circ$), muscovite ($2\theta = 8.9^\circ$), and pyrite ($2\theta = 56.5^\circ$). Sample C shows higher counts of quartz and albite than Sample A or B. Smectite group was not detected.

X-ray counts of albite and chlorite–serpentine increase by soaking, whereas those of orthoclase decrease after soaking. The effects of milling and stirring were

Table 3. XRF data shows the concentrations of the 10 major elements (Na_2O , MgO , Al_2O_3 , SiO_2 , P_2O_5 , K_2O , CaO , TiO_2 , MnO , and Fe_2O_3). The values are corrected by multiplying $(100-\text{LOI})/100$ to include volatile components, where LOI is the loss on ignition.

Sample Name	Na_2O mass%	MgO mass%	Al_2O_3 mass%	SiO_2 mass%	P_2O_5 mass%	K_2O mass%	CaO mass%	TiO_2 mass%	MnO mass%	Fe_2O_3 mass%	LOI %	Total mass%
A-1	2.155	2.138	15.268	56.785	0.154	4.040	0.876	0.731	0.124	6.909	9.914	99.094
A-2	2.104	2.304	15.471	58.084	0.194	3.197	1.028	0.743	0.134	7.176	8.541	98.977
A-3	2.312	2.289	15.415	57.938	0.169	3.154	1.023	0.743	0.142	7.198	8.545	98.928
A-4	2.180	2.450	15.649	58.138	0.163	2.831	1.025	0.748	0.138	7.218	8.752	99.292
B-1	2.186	2.120	15.060	56.578	0.163	4.031	0.933	0.721	0.135	7.084	10.157	99.168
B-2	2.140	2.313	15.480	58.095	0.160	3.187	1.003	0.747	0.138	7.213	8.543	99.020
B-3	2.364	2.326	15.611	57.857	0.158	3.073	0.978	0.744	0.129	7.042	8.827	99.108
B-4	2.239	2.401	15.425	57.793	0.167	2.841	0.997	0.743	0.139	7.186	8.951	98.881
C-1	2.377	1.774	14.284	61.674	0.110	4.004	0.869	0.594	0.057	5.424	8.134	99.301
C-2	2.182	1.815	14.196	63.381	0.102	3.292	1.004	0.599	0.065	5.594	6.914	99.145
C-3	2.505	1.903	13.955	63.382	0.116	3.073	1.131	0.584	0.063	5.517	6.815	99.047
C-4	2.330	1.979	14.300	63.253	0.112	2.954	0.940	0.612	0.067	5.621	6.941	99.109
Error (1σ)	0.037	0.062	0.010	0.037	0.002	0.019	0.017	0.010	0.002	0.013	0.283	0.296

inconsistent. After soaking, pyrite tends to be washed away to below the detection limit.

4.4 Seawater

Table 6 presents the Na^+ , K^+ , Mg^{2+} , Ca^{2+} , Cl^- , and SO_4^{2-} concentrations in seawater sampled before and after soaking. Increased K^+ concentration after soaking agrees with the decrease in K_2O in XRF data. The Na^+ ,

Cl^- , and SO_4^{2-} concentrations also showed minor increases after soaking.

Relations between soaking time and the differences among Na^+ , K^+ , and Cl^- concentrations are presented in Fig. 2. Dissolution progressed with time, particularly in the stirred samples. However ΔCl^- decreased after 6 h.

Table 4. The contents of total carbon (TC), total nitrogen (TN), total sulfur (TS), inorganic carbon (IC), and total organic carbon (TOC). TOC is calculated by subtracting the IC from the TC.

Sample Name	Total carbon (wt %)	Total nitrogen (wt %)	Total sulfur (wt %)	Inorganic carbon (wt %)	Total organic carbon (wt %)
A-1	3.187	0.080	0.115	0.431	2.756
A-2	2.372	0.077	0.085	0.450	1.922
A-3	2.268	0.076	0.097	0.468	1.800
A-4	2.356	0.076	0.091	0.452	1.903
B-1	3.193	0.076	0.104	0.473	2.719
B-2	2.353	0.076	0.082	0.478	1.875
B-3	2.290	0.074	0.096	0.432	1.858
B-4	2.501	0.075	0.085	0.459	2.042
C-1	2.466	0.056	0.209	0.401	2.065
C-2	1.890	0.054	0.149	0.462	1.428
C-3	1.615	0.054	0.144	0.475	1.141
C-4	1.832	0.057	0.217	0.410	1.422
Error (1σ)	0.020	0.004	0.050	0.009	0.022

Table 5. X-ray counts for representative minerals from XRD data.

Sample Name	Quartz Height (cps)	Albite Height (cps)	Orthoclase Height (cps)	Chlorite-Serpentine Height (cps)	Muscovite Height (cps)	Pyrite Height (cps)
A-1	1688	280	132	33	19	15
A-2	1675	338	97	40	17	n/d
A-3	1697	244	118	36	13	n/d
A-4	1723	233	94	42	12	n/d
B-1	1625	225	142	31	19	20
B-2	1655	262	114	33	13	n/d
B-3	1644	289	125	41	17	33
B-4	1803	251	124	38	19	n/d
C-1	2453	332	120	24	17	25
C-2	2694	448	76	31	25	n/d
C-3	2633	475	107	27	27	n/d
C-4	2565	419	76	33	22	22

Table 6. The Na^+ , K^+ , Mg^{2+} , Ca^{2+} , Cl^- , and SO_4^{2-} concentrations in seawater sampled before and after soaking.

Sample Name	Na^+ (mM)	K^+ (mM)	Mg^{2+} (mM)	Ca^{2+} (mM)	Cl^- (mM)	SO_4^{2-} (mM)
A-Seawater	454.7	9.8	51.0	10.0	532.1	26.5
A-2	461.9	18.3	50.9	9.8	541.2	27.0
A-3	457.0	18.6	50.5	9.7	541.1	26.9
A-4	465.6	23.5	50.5	9.7	543.5	27.0
B-Seawater	446.7	9.7	50.2	9.8	540.0	26.8
B-2	463.3	36.4	52.3	9.8	547.3	27.3
B-3	460.4	32.0	51.0	9.6	547.4	27.3
B-4	481.1	34.5	52.2	9.9	566.8	28.2
C-Seawater	456.9	9.9	51.5	10.1	528.4	26.2
C-2	466.4	19.0	51.3	9.9	544.7	27.0
C-3	457.4	17.6	50.2	9.7	534.5	26.6
C-4	460.2	26.8	52.6	10.1	552.0	27.4
Error (1σ)	2.7	0.1	0.3	0.06	2.7	0.1

5. Discussion

Major element analyses reveal that Na_2O and K_2O contents of the cuttings decrease by soaking. Considering the order of magnitude difference between drilling mud

and seawater, we infer that changes in K contents reflect the removal of drilling mud from cuttings. Changes in Na contents also provide supportive data. However, most other element concentrations, such as those of Mg, Al, Si, Ca, Ti, Mn, and Fe, showed slight increases, suggesting an inverse relation that is dominated by decreased Na and K.

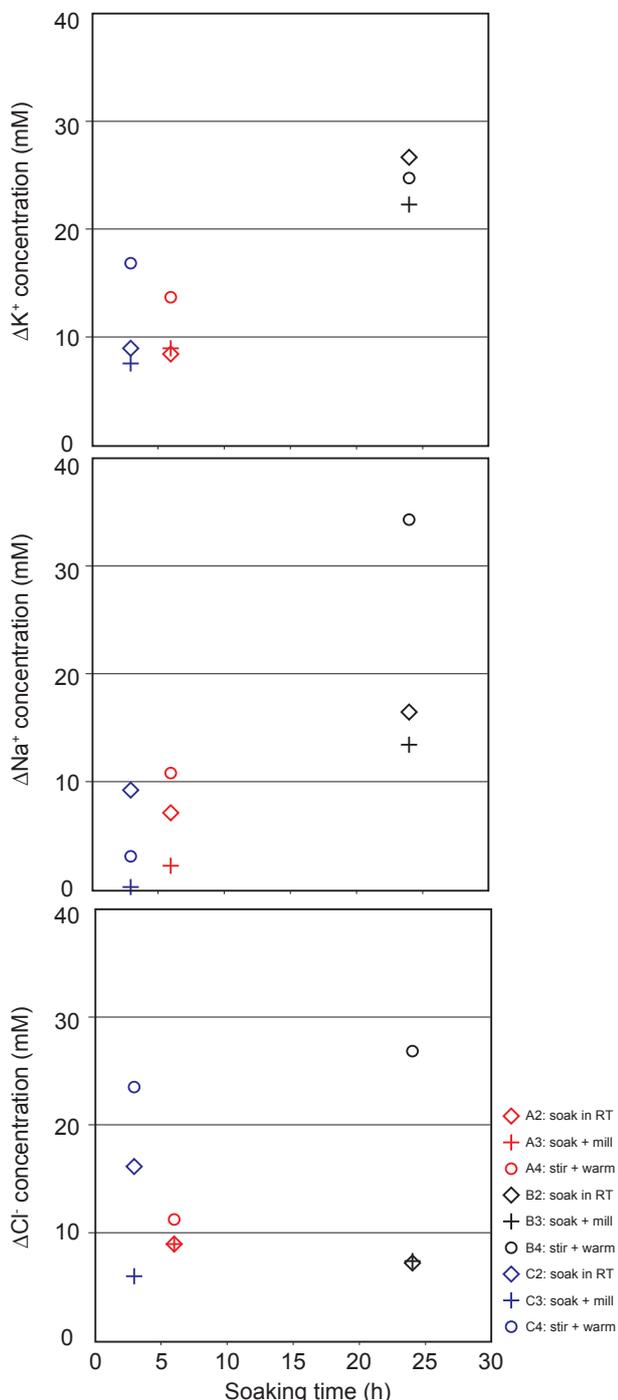


Fig. 2. Relations between soaking time and the difference (Δ) between concentrations of K^+ (top), Na^+ (middle), and Cl^- (bottom) in seawater after the soaking of the cuttings into seawater and those immediately before starting the soak. Soaking time of sample group A is 6 hours, sample group B is 24 hours, and sample group C is 3 hours.

5.1 Dissolution of KCl from cuttings

To minimize the effects of inter-elemental variations, K_2O content is divided by TiO_2 contents (Fig. 3). The $\text{K}_2\text{O}/\text{TiO}_2$ ratios in XRF data decrease and the K^+ concentration in seawater increased after the soaking, indicating that KCl is dissolved during soaking. All samples show a decrease of $\text{K}_2\text{O}/\text{TiO}_2$. Subsamples 4 (stirring) exhibited the greatest decrease.

Figure 3 shows that $\text{K}_2\text{O}/\text{TiO}_2$ ratios exhibited little change with soaking time between 6 h and 24 h. Although the differences between 3 h and 6 h might be attributable to different lithology of the samples, the data indicate that the soaking time of 6 h was sufficient to reach equilibrium.

5.2 Dissolution of NaCl from cuttings

Figure 4 shows that $\text{Na}_2\text{O}/\text{TiO}_2$ ratios decrease by the soaking, with or without stirring. When the cuttings are milled before soaking, the $\text{Na}_2\text{O}/\text{TiO}_2$ ratios increase after the soaking (Subsample 3). The Na^+ in seawater increased in all cases, but the rate of increase was minimal in the case of the milled samples. These results suggest that Na^+ was absorbed on the increased surface of the milled cuttings. The absorption is possible only if the inside of the cuttings have lower Na content than the original surface because, otherwise, the ratio of dissolution versus absorption would be the same whether or not the samples were milled. This fact supports the hypothesis that the dissolved Na^+ originates from drilling mud that remained on the cutting surfaces.

Similarly to the case of $\text{K}_2\text{O}/\text{TiO}_2$ presented in Fig. 3, Fig. 4 shows that $\text{Na}_2\text{O}/\text{TiO}_2$ ratios exhibited only slight differences between the soaking times of 6 h and 24 h. That fact confirms that the soaking time of 6 h was sufficient to reach equilibrium.

5.3 Contamination of organic material and removal from cuttings

The LOI and TOC contents of the cuttings decrease after soaking, suggesting that water removed organic materials (Figs. 5 and 6). Sample C3 showed a notable

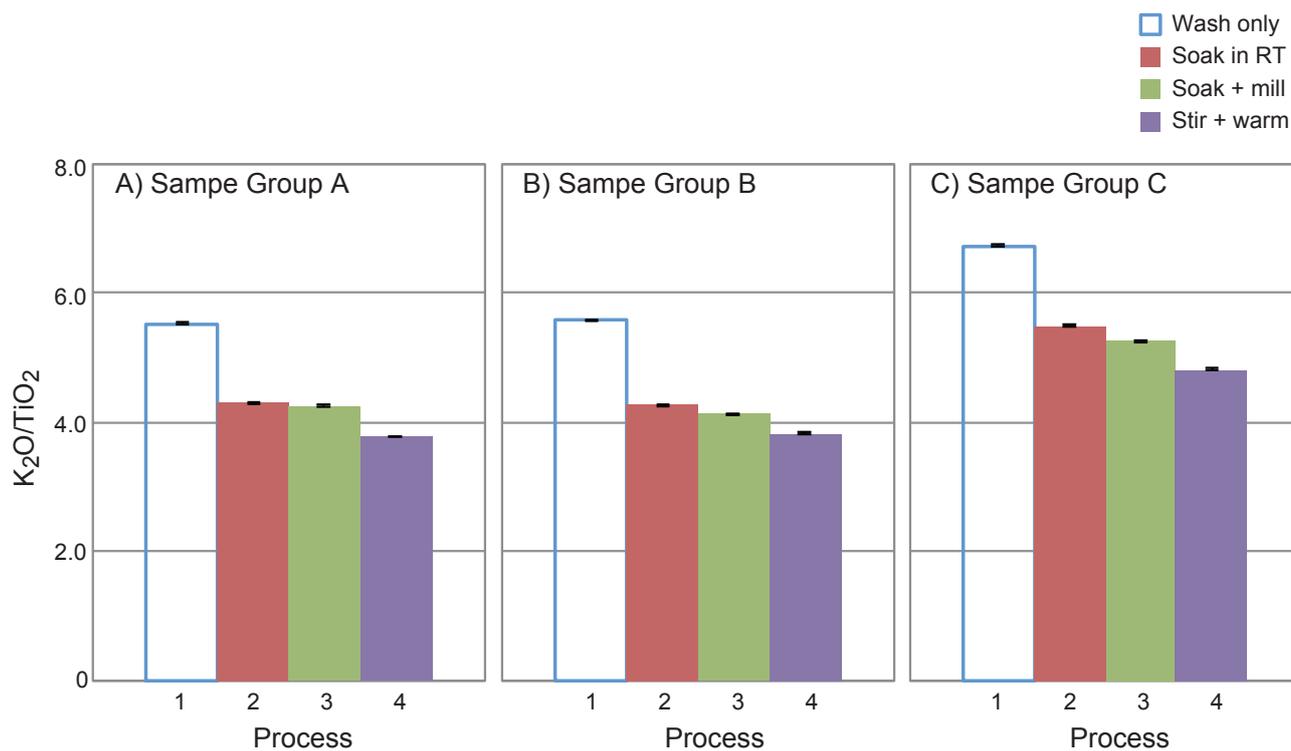


Fig. 3. Content ratios of K₂O/TiO₂ in cuttings decreased after soaking, but showed little difference between 6h (sample group A, left) and 24h (sample group B, middle).

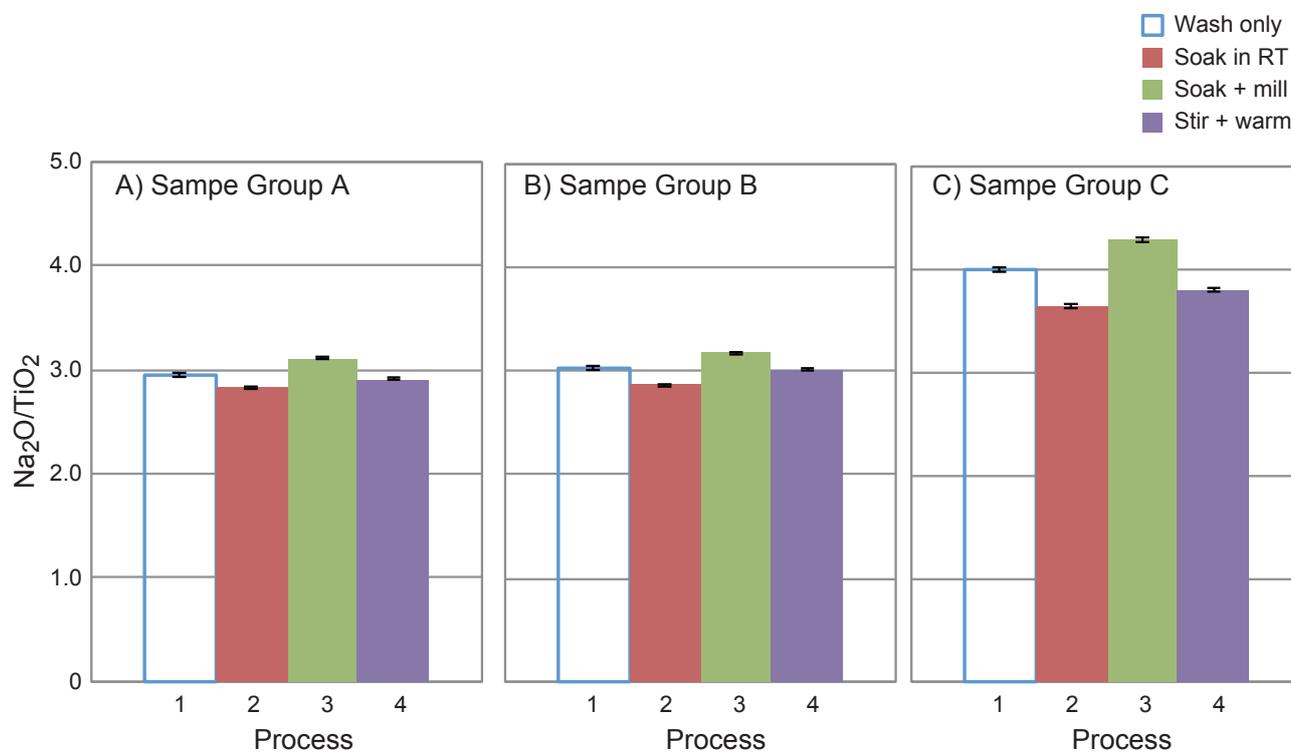


Fig. 4. Content ratios of Na₂O/TiO₂ in cuttings decreased after soaking, but showed little difference between 6h (sample group A, left) and 24h (sample group B, middle).

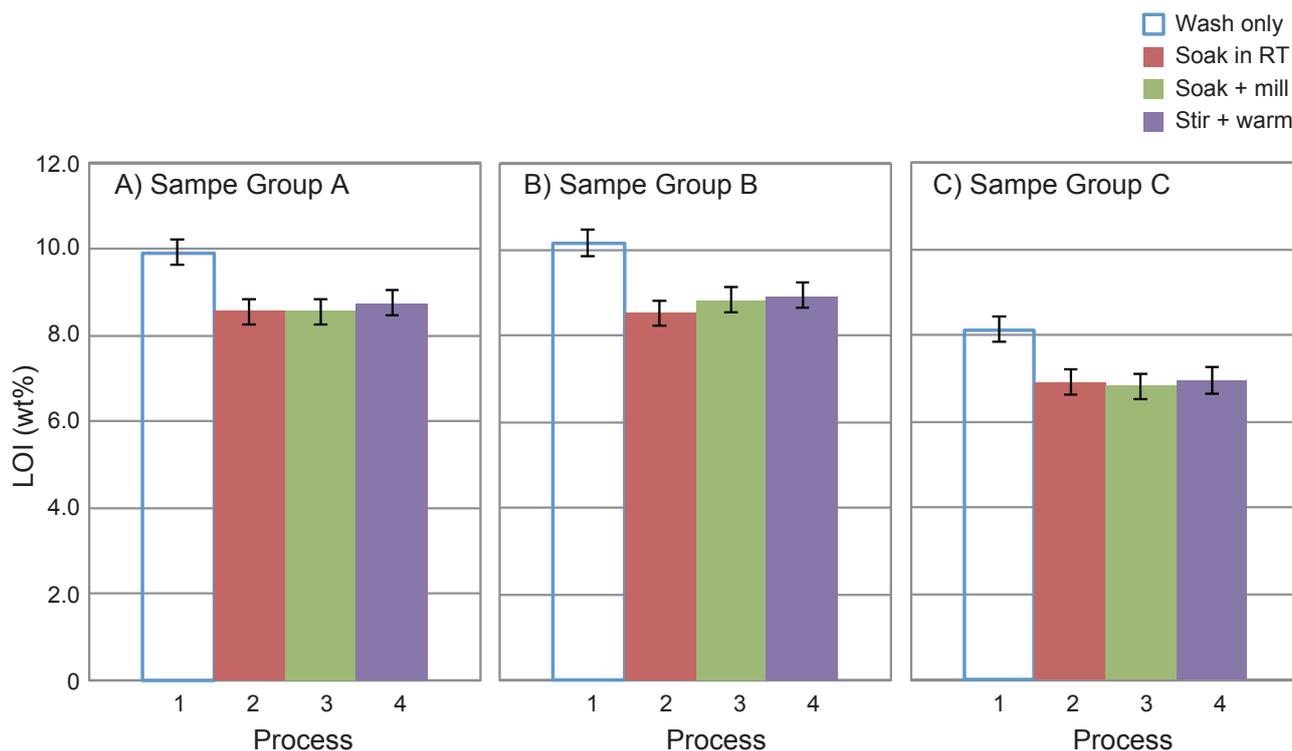


Fig. 5. LOI in cuttings decreased by soaking, but showed slight effects from milling and stirring.

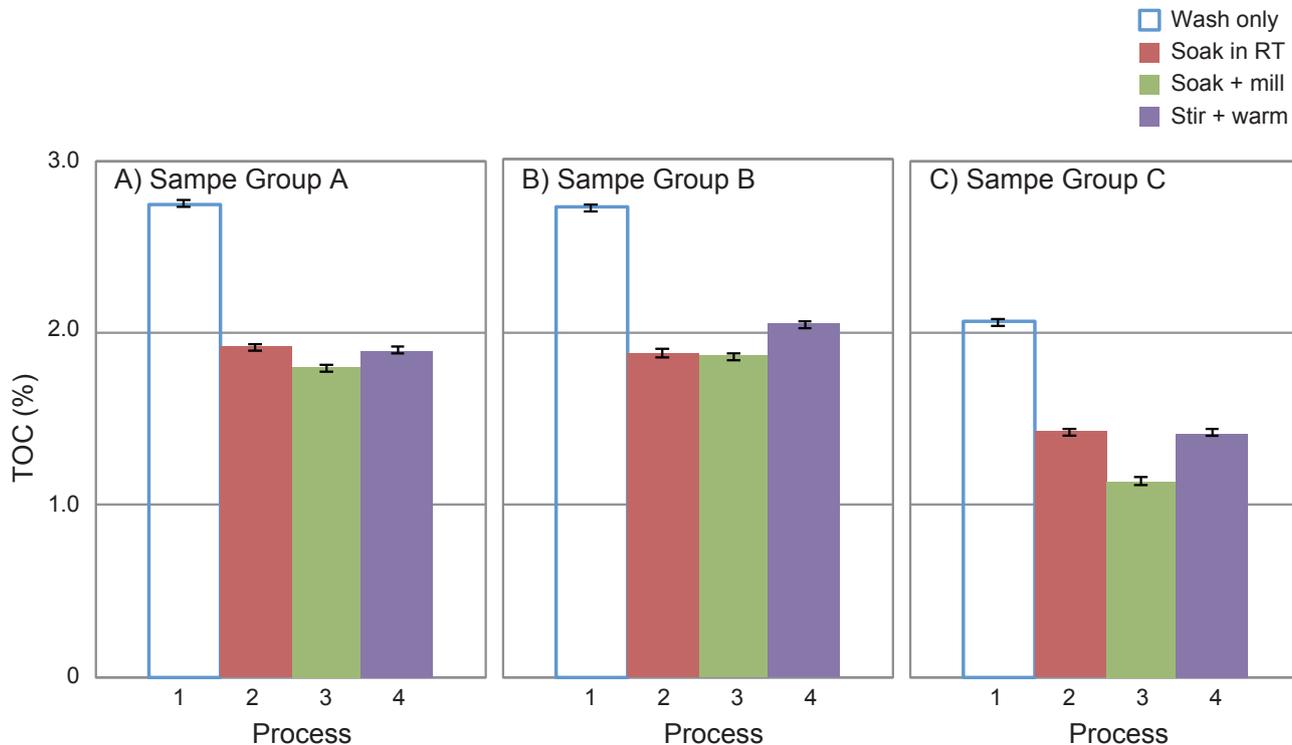


Fig. 6. TOC in cuttings decreased by soaking but showed slight differences between soaking time of 6 h (sample group A, left) and 24 h (sample group B, middle).

decrease in TOC, but it remains unclear whether the removed organic carbon originated from the drilling mud or from the cuttings. Slight differences were found between 6 h and 24 h soaking times. Effects on milling or stirring were minor and inconsistent.

5.4 Variation of mineral compositions by soaking

Figure 7 shows X-ray count ratios of (top) orthoclase/quartz and (bottom) chlorite-serpentine/quartz. The rate of decrease of orthoclase/quartz by soaking with seawater might reflect the orthoclase hydrolysis during

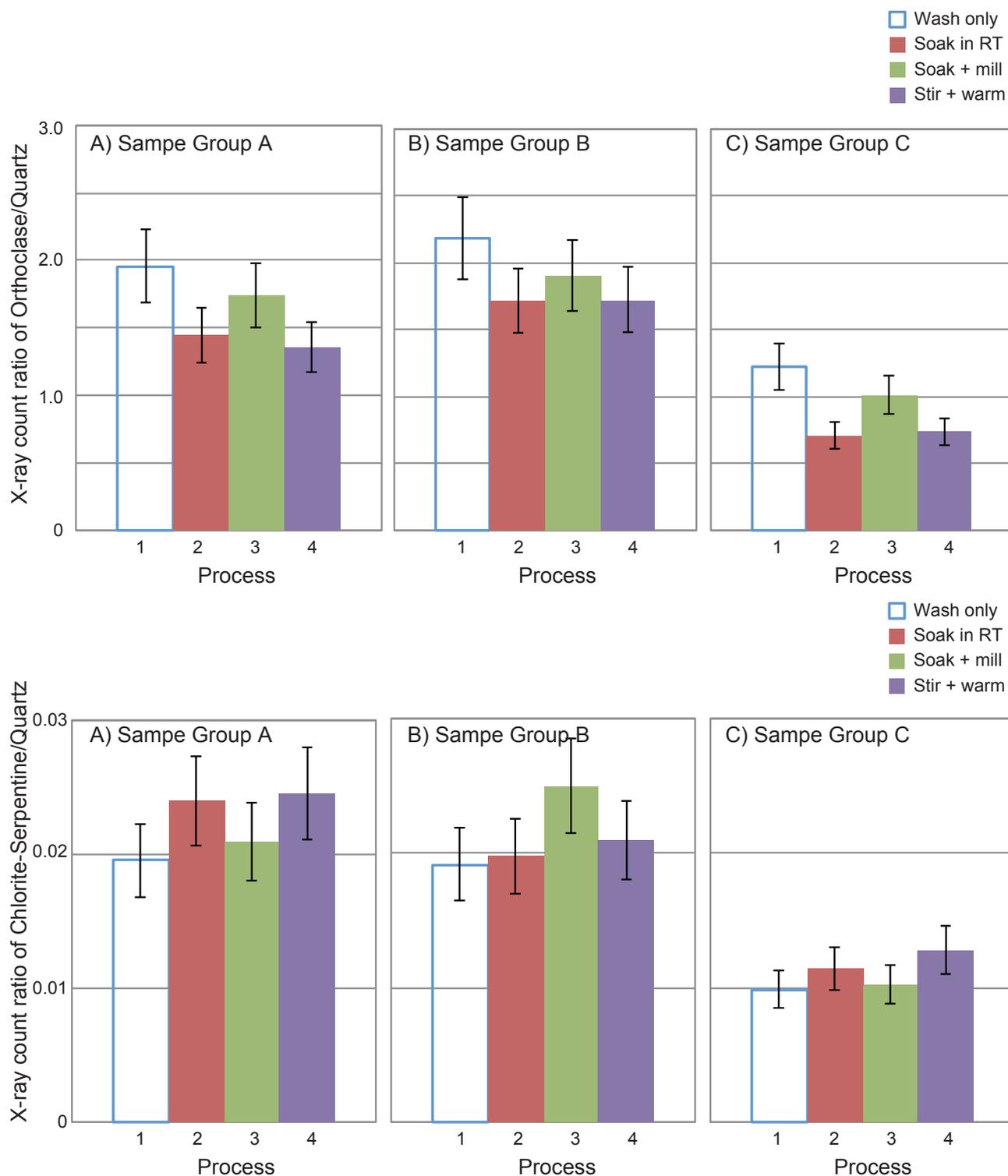


Fig. 7. X-ray count ratios of orthoclase/quartz (top) and chlorite-serpentine/quartz (bottom).

soaking, but further analyses must elucidate the clay mineral behavior.

5.5 Proposed cutting treatment to reduce drilling mud effects

Our results demonstrate that K_2O/TiO_2 ratios decrease more than 30% by soaking in heated seawater at 40°C for 6 h. Furthermore, the Na_2O/TiO_2 ratios decrease by soaking with seawater, but the rate of decrease is less than 10% when the sample is not milled. Moreover, TOC and TS contents decrease by soaking with or without heating and stirring. These results suggest that stirring of cuttings in heated seawater at 40°C for 6 h after quick washing is effective for XRF and TOC analyses. Further experiments can assess the treatments and suggest the best protocol.

Acknowledgements

The authors thank two reviewers, Drs. Sanny Saito and Wataru Tanikawa, for their constructive comments. This research used samples provided by the Integrated Ocean Drilling Program (IODP).

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