Amorphous silica extraction procedure for Site U1480, Expedition 362

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The silica extraction procedure we employed is based on the one described by Lyle and Lyle (2002), which was designed to quantify biogenic silica in radiolarian rich Eocene sediment. As described by these authors, the commonly used 2M Na2CO3 extraction solution does not fully dissolve the biogenic opal in Eocene-Paleocene samples, because these fossil assemblages are larger and thicker than the Neogene opal counterparts; instead, Lyle and Lyle (2002) recommend the use of a 2M KOH solution. This reagent dissolves biogenic opal as well as volcanic glass and other amorphous silica phases, but does not dissolve significant amounts of SiO2 from clay minerals or quartz (Lyle and Lyle, 2002).

To determine the amorphous silica content at Site U1480, we weighed 30 mg of freezed-dried and ground sediment sample and placed it in a 50-mL Corning CentriStarTM centrifuge tube to which 20 mL 2M KOH was added. Tubes were capped, mixed for 30 seconds with a vortex mixer and placed in a water bath at 85oC. Lyle and Lyle (2002) recommend a 10 hr extraction, but since we did not have a priori knowledge of the behavior of the amorphous silica phases in our samples, we collected aliquots of the extract liquid at various time intervals (from 5 min to 24 hrs) after the digestion began. The samples were vortex mixed after each aliquot was drawn. The extract liquid was analyzed for dissolved silica using the method based on the production of a yellow silicomolybdate complex, by drawing 10-200 uL aliquots after the solution cooled. The silicomolybdate complex was reduced by ascorbic acid to form molybdenum blue (Grasshoff et al., 1983), and measured at 812 nm with an Agilent Technologies Cary Series 100 UV-Vis spectrophotometer. Results from replicate (n=2, 3) analyses of two samples from different lithologic units yielded concentrations that agreed within 2% in all the leaching steps.

Results from the extractions are listed in Table SM-Chem-1. Figure SM-CHEM-1 shows the amount of silica leached as a function of time for all samples analyzed, according to their lithologic Unit (see “Site U1480 Sedimentology and Petrology”). Results of these analyses are discussed in Hüpers, et al., in press.

References

Hüpers, A., et al., in press, Release of mineral-bound water prior to subduction tied to shallow seismogenic slip off Sumatra, Science.

Grasshoff, K., Ehrhardt, M., and Kemling, K. (eds.), 1983. Methods of Seawater Analysis: Weinheim, Germany (Verlag Chemie Int.).

Lyle, A.O. and Lyle, M. W. 2002. Determination of biogenic opal in pelagic marine sediments: A simple method revisited. In: Lyle, M., Wilson, P.A., Janecek, T.R., Proceedings of the Ocean Drilling Program, Initial Reports Volume 199: College Station, TX (Ocean Drilling Program).

Figure caption: Silica released as a function of extraction time, expressed as sediment weight percent. Samples analyzed are separated according to their lithologic Unit.

Table 1. Data listing of silica concentrations measured in the KOH extracting solution at various intervals ranging from 5 min to 24 hours.