TECHNICAL NOTE

A NEW METHOD OF FOSSIL PREPARATION, USING HIGH-VOLTAGE ELECTRIC PULSES

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INTRODUCTION

The availability of a complete and efficient method for separating a rock's constituents is of interest to a wide spectrum of earth scientists. They range from those interested in large-scale ore processing to mineralogists or geochemists wishing to separate single crystals for crystal-structure determinations or isotopic measurements and to paleontologists wishing to extract fossils from a variety of rock types. Physically disaggregating well-lithified sedimentary rocks to recover minerals or fossils is hampered by breakage or production of multimineralic fragments (composite grains) during crushing. Selective dissolution of whole rocks using acids to separate soluble and insoluble components is not always effective.

Hutchison (1974) reviewed techniques for crushing whole rock specimens to achieve good mineral separation. He stated that care must be taken not to crush the rock too finely but enough to ensure that the fragments will be monomineralic. He also noted that the rock dust released during the crushing is a health hazard and that it must be cleaned from the surfaces of mineral grains before magnetic or heavy liquid separation. Crushing commonly produces multimineralic grains, which hampers efficient separation of different minerals. Kummel and Raup (1965) described paleontological preparation methods, including heat treatment, miniature sand blasting, and dissolution. For the latter method, the sample might need to be soaked for many days, and care must be taken that the specimen itself is not lost to dissolution.

Over the past few decades, the feasibility of Electric Pulse Disaggregation (EPD) for mineral separation has been investigated by researchers in the former Soviet Union (Maurer, 1968; Kurets et al., 1989; Finkelshtein et al., 1989; Shuloyakov et al., 1989; Rudashevsky et al., 1995a), Europe (Andres, 1977, 1989; Andres and Bialecki, 1986; Bialecki et al., 1992; Saini-Eidukat et al., 1993), and the U.S. (Touryan et al., 1992; Weiblen, 1994; Rudashevsky et al., 1995b). EPD uses high-voltage pulses to fragment a rock sample into its constituents.

These independent investigations have shown that EPD has several advantages over conventional crushing and grinding methods. These include: (a) breaking of minerals into their natural size distribution, as present in the rock and regardless of

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the grain size; (b) lower production of rock dust; and (c) formation of fewer multi-mineralic grains. Disadvantages include gaining access to specialized equipment not yet commercially available, the use of high voltage, and the need for the sample to be immersed in or saturated with a suitable dielectric liquid such as water or oil.

THE EPD INSTRUMENT

The basic components of the unit are a high-voltage power source fed to a voltage-increasing circuit, which delivers a high-voltage discharge to the sample chamber (Figure 1). With our instrument, the high-voltage power source is a Phillips X-ray generator (with X-ray tube removed). The voltage-increasing circuit consists of a number of capacitors, known as a Marx circuit. The capacitors are charged in parallel through inductance coils and discharged in series across spark gaps. The working instrument at the University of Minnesota uses a 50 kV power supply and contains three 100 kV, 0.1 μF capacitors. The water bath in which the sample rests actually acts as the last capacitor in the system, and the entire apparatus is enclosed in a Faraday cage. When 40 kV is supplied to the two capacitors, a pulse of 120 kV is delivered to the sample chamber at a rate of approximately one pulse per second.

At voltages below approximately 100 kV, the fluid in which the sample is immersed electrically breaks down before the rock and the electrons circumvent the sample. At higher voltages, however, the rock electrically breaks down before the fluid, apparently preferentially along zones of weakness in the solid. In the case of rocks, these zones of weakness are apparently grain boundaries between mineral phases with different dielectric constants.

Electric-pulse disaggregation is not a completely nondestructive process; a less than one percent fraction of spheres or fragments of melted or spalled electrode material, (i.e., steel, copper, or aluminum), is found in the disaggregated material.

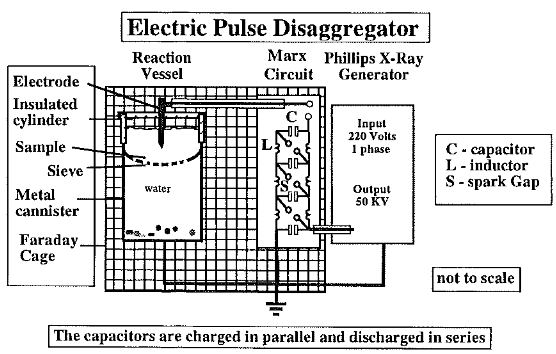

FIGURE 1. Diagram of the EPD. Schematic drawing of the electric pulse disaggregator. The instrument consists of four components: a high-voltage power source, a Marx circuit, a reaction vessel, and a Faraday cage. For our instrument, the high-voltage power source is a Phillips X-ray generator. The Marx circuit is described in the text. The reaction vessel in our instrument is a 10-cm-diameter stainless steel canister, in which a hemispherical sieve (3 mm sieve size) is inserted, both of which are grounded. The top of the reaction vessel consists of a polyvinyl cover that supports the stainless-steel electrode.

The spheres are apparently formed by melting of the sample and are generally fine-grained (1–100 µm).

APPLICATIONS IN PALEONTOLOGY

The first applications of EPD were devoted to mineral extraction. We were pleasantly surprised to discover that it also is a very effective method for extracting fossils. Here we present examples of EPD experiments made on fossiliferous sediments from North Dakota and South Dakota. Run products were dried, size classified, and examined, using optical and scanning electron microscopy.

The first example is from the Carlile shale (Upper Cretaceous). These shallow inland sea sediments are exposed as a thin veneer lying unconformably on Precambrian granites in quarries of northeastern South Dakota. The Milbank site probably represents a bedrock high in the Cretaceous sea, which formed islands or steep shorelines. Our samples are from the Cold Spring granite quarry near the town of Milbank (outcrop B of Merewether [1983], sec. 18, T. 120 N., R 46 W., Grant County). There is a conglomerate at the base of the shale that includes pieces of weathered granite, belemnites, shark teeth, and other components all cemented by carbonate.

We originally attempted EPD on these rocks to separate the abundant shark teeth. Some teeth were successfully freed, but the method charred and broke many of the larger specimens. However, EPD successfully liberated large numbers of microfossils, which were excellently preserved as pyrite molds.

EPD liberated a diverse and large number of microfossils from samples from this location (Figure 2). Sloan (1964) reported similar pyrite molds of microfossils recovered using acetic acid dissolution. No post-EPD cleaning was done on these samples; even so, the separation from matrix and preservation of surface detail is excellent. Mineral overgrowths on the ostracode molds obscure details and may make precise identification difficult (F. Swain, personal communication, 1994).

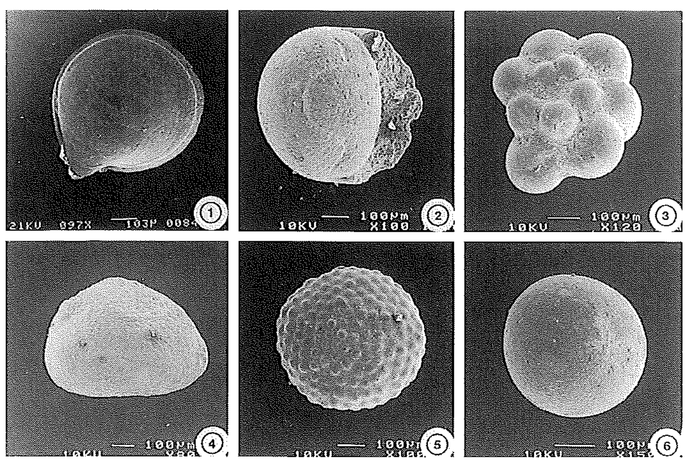

FIGURE 2. Scanning electron micrographs of microfossils recovered from the Carlile shale at Milbank, South Dakota. 1. juvenile clam; 2. ammonitella; 3. foraminifera; 4. ostracode; 5. spheroid; 6. spheroid.

The observation that ammonitellas found at this site (Figure 2.2) match the size of those found elsewhere supports the idea that ammonite hatchlings were planktonic as opposed to the larger hatchlings of contemporaneous nautiloids (about 12 mm) that probably swam in deeper water (Landman, 1984; N.H. Landman, personal communication, 1995). The large numbers of ammonitellas found support Landman's idea that the reproductive strategy of ammonites was to release large numbers of eggs as opposed to the strategy of modern nautiloids of laying 5 or 6 eggs per year. Sedimentologically, this outcrop is interesting because there are nonpyritized fossils (belemnites, shark teeth) mixed in with pyritized fossils in a high-energy environment. This observation could imply that the microfossils were pyritized elsewhere and transported to this site.

Figures 2.5 and 2.6 show spheroidal objects recovered from the Milbank basal conglomerate. Hundreds of these were collected from the fine fraction of the EPD sample and separated using a Frantz magnetic separator. The spheroids are intimately associated with the pyrite microfossil molds; some have smooth surfaces (Figure 2.6), while others have ornamented surfaces (Fig. 2.5). They do not appear to be artifacts such as melted pyritized fossil molds or melted pyrite concretions because they can be observed *in situ*. Also, casts of spheroids are observed in the calcite matrix. The origin of these spheroidal objects is enigmatic. Their approximate diameter is 300–500 μm, an order of magnitude larger than typical framboids reported in, e.g., Rickard (1970) or Sawlowicz (1993). Whether they are biogenic, perhaps megaspores, or deposited by geochemical processes is equivocal and awaits further characterization.

Other fossiliferous rocks to which we have applied EPD include the Minnelusa Fm (Pennsylvanian-Permian) sampled from Elk Creek, 12 miles N. of Rapid City, South Dakota (Allan Ashworth and Joe Martinetti, collectors). This rock is a fine-grained sandstone, from which we recovered conodont fragments (Figure 3). A third sample is from a drill core of the Mowry Formation (Upper Cretaceous) from a well in Burleigh County, North Dakota (John Hoganson, collector). Macrofossils that were recovered include a belemnite tip, fish scales and fish teeth. EPD also preserved the cast of a fish scale, which might have been destroyed in conventional processing.

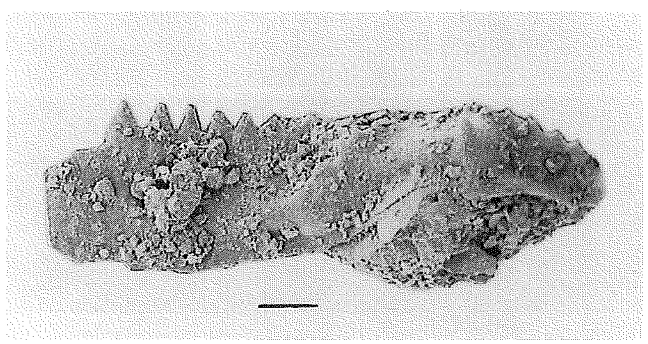

FIGURE 3. A conodont fragment from the Minnelusa Fm (Pennsylvanian-Permian). Scale bar represents 100 μm.

Further experiments were made on a Cretaceous age tar sand and showed that nautiloids can be cleanly liberated from the asphalt mineralized matrix. The largest fossils separated to date are teeth or ammonites in the centimeter size range.

CONCLUSIONS

1. For the samples in this study, EPD allowed us to separate hundreds of microfossil molds quickly, efficiently, and cleanly. Perhaps the greatest advantage of EPD is that the separation usually occurs along natural boundaries, resulting in whole, unbroken components. This pattern of breakage allows petrologists, geochemists, mineral engineers, and paleontologists to examine items of interest recovered from the rock with minimal damage and with surface features preserved.

2. Although we have demonstrated that EPD can be used as a method of fossil liberation, its suitability for a particular sample must be determined on an individual-case basis. A well-defined interface between minerals of contrasting dielectric constant should be present for the physical process to operate. If such an interface is not present (e.g., in recrystallized limestones), breakage across fossils may occur.

3. The high-voltage apparatus can be lethal, and instrument operation must be conducted with great care. Anyone contemplating the construction of an EPD apparatus is encouraged to contact the authors. For EPD to become a routine fossil or mineral-separation method, considerable method development is required.

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