The University of Wisconsin-Stevens Point was recently asked by the Arizona Museum of Natural History (AZMNH) to analyze eleven late Cretaceous sedimentary rock samples from the Fort Crittenden Formation in southeastern Arizona for fossil pollen and other palynomorphs (Table 1). The Fort Crittenden, well-known for its Campanian faunal assemblages, outcrops in the Santa Rita Mountains near Tucson (Figure 1). In the spring of 2014, researchers affiliated with the AZMNH collected the samples from layers that harbor dinosaur and other vertebrate remains. The Fort Crittenden members are comprised of conglomerates, shales, and sandstones that are interpreted to be freshwater (fluvial and lacustrine) and subaerially-derived valley deposits. Despite sample fossiliferous lithologies, the rock is not fossilized wood. Little is known of the flora or the micropalaeontology. To better understand the environments in which these dinosaur remains accumulate, we are using physical and chemical preparation techniques to disaggregate the rock and identify palynomorphs. Light and scanning electron microscopy are being used for pollen identification. Experimental use of x-ray diffraction to detect cellulose presence will be tested for pollen recognition. Preliminary results show a high degree of sediment oxidation with several possible pollen and non-pollen palynomorphs that may be useful paleoenvironmental indicators.

**Methods**

Subsamples of the rocks were crushed into sand sized particles. Then the particles were disaggregated in distilled water and 4 ml of 10% HCl. After this was done, the mixture was tested for effervescence and the color was recorded. Next, additional 10% HCl acid was added until effervescence was completed. After washing, 10 ml of KOH was added to the samples to remove organic acids. Samples were again washed and test slides made from each sample after this step. To dissolve silicates, hydrofluoric acid was added to the samples and the samples placed in a hot bath. Following HCl, the samples were treated with HCl to remove colloidal silica precipitates, then decanted and followed by a double washing with distilled water. Next, Sodium Metaphosphate (SMP) was used to digest clays not removed by HCl. Finally, the samples were centrifuged at low speed and the clays decanted. After a final wash, tertiary butyl alcohol (TBA) was added to dehydrate the samples and silica oil was added to each vial and heated at low temperature to evaporate the remaining water. Additional silicon oil was added as needed to each sample for slide preparation.

Sample AC04, a black shale, was found to be very rich in organic substances and additional steps were needed to dissolve suspected kerogen and other organic matter. AC04 was split into two samples in which one was additionally rehydrated with KOH and hot H2O2. These additional steps did appear to lighten the sample slightly but did not clearly remove enough to reveal more pollen.

Before preservation in silicon oil, the samples were taken for SEM analysis. Using a vacuum tube and non-conductive adhesive discs, a drop of each sample was glued to a 10 mm platform. The samples were then allowed to dry and placed in the Scanning Electron Microscope for analysis. The SEM was calibrated and the Environmental Scanning Electron Diffraction for observation. Only sample LC01 has been analyzed to date.

**Results**

Findings were sparse using light microscopy. Isolated pollen grains were found in AC01, AC01B, AC04, and LC01. AC01 (Figure 2) presented an insignificant sample of Vigna pollen grain. ACO2 (Figure 3) had a more obvious and well intact Pinus grain along with an undifferentiable round palynomorph. ACO2 (Figure 4) presented a well preserved Chenoepogon grain. Lastly, LC02 (Figure 5) presented a non-pollen microfossil and a cf. Glomus spore with hyphal attachment (Van Geel, 2001). We see ‘shadows’ of what appear to be palynomorphs in some samples, but the grains are too oxidized or degraded to identify. We have seen plant and insect parts which are well preserved, but there is nothing sufficiently diagnostic to be certain if they are Campanian in age. AC04 (black shale) contains a lot of amorphous mass. There are definite remnants of what look like trilite spores in AC04, but they are eroded beyond recognition. In this case it seems that oxidation of the organic matter (and pollen) probably happened at or not long after deposition, possibly by fire. Although no obvious charcoal is preserved.

The most likely explanation for a lack of palynomorphs, or the highly degraded grains we did observe, relates to the collection of the samples. We do not have much information about the field collection of the samples, but the highly weathered nature of the rock suggests they came from, or very close to, the surface. Proper collection as described by Finkelshtein (2005) indicates rock samples should be collected from a depth of 40-80 cm to avoid surface oxidation and contamination. Also, as cited by Traverse (2007), the likelihood of even finding a statistically significant number of grains (200 per slide) decreases dramatically with smaller beginning samples. Due to the limited amount of rock we received, we had to use a sample size of 0.5 grams, whereas most procedures call for upwards of 5 grams. Lastly, there is evidence that some of the Fort Crittenden strata were wash layers deeply impacted by the constant presence of forest fires (Finkelshtein et al. 2005). These channel environments (Finkelshtein et al. 2005) were also likely to have been constantly changing and probably did not preserve pollen well.

**Conclusions**

Only five of the samples contained any identifiable pollen, and the strong preservation of these grains suggests they are more recent compared to the samples in some samples. This indicates strong weathering or oxidation of the microfossiliferous material. The collection of surface rocks instead of deeper samples is because we are looking for a possible indication of finding large pollen or palynomorphs while also increasing the likelihood of contaminants present in the sample. Future attempts at palynology on these shale will require sampling below any surface weathering and inclusion of larger samples sizes.

**References**


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