On the use of some phosphates in the preparation of ostracod shells

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ABSTRACT - Common preparation techniques for ostracods include the use of water-softeners containing sodium hexametaphosphate and/or sodium tripolyphosphate, to disaggregate sediments. Here, ostracod shells were treated with phosphatic water-softener in tap and distilled water. Concentrations as low as 2.5% in as little as six hours caused significant damage. The worst damage occurred in concentrations of water-softener at the middle of the range used (0 – 20%). Alteration could be misinterpreted as taphonomic. Taxa are not equally resistant and assemblages could be altered, leading to erroneous conclusions about diversity, dominance, and differential transport.

INTRODUCTION
In recent years there has been little mention of preparation techniques in studies of Ostracoda, although it has been long recognized that such methods can alter shells. Common practice involves the use of sodium hexametaphosphate [(NaPO₄)₆] (Jones, 1956) and/or sodium tripolyphosphate (Na₅P₃O₁₀). They are marketed in the U.S.A. in the product “Calgon Water Softener”, for example. Water softener is dissolved in water to clean shells and disaggregate sediments. Here we report on the effects of the phosphates on ostracod shells, as commonly used by North American ostracodologists.

MATERIAL AND METHODS
Living species were used from the Louisiana Continental Shelf (Kontrovitz, 1976) while fossils were obtained from Pleistocene sediments from Florida (Kontrovitz, 1978) and Eocene deposits from Louisiana (Kilmartin, 1982). Unweathered shells were selected from modern specimens (Oertli, 1975) and undamaged fossils were chosen (Table 1).

Waters from five sources were used along and with Calgon (Table 2). Untreated adult and juveniles (washed only with tap water) were immersed in glass vessels with each water type and 0, 2.5, 5, 10, 15, and 20% of Calgon, by weight. Trials were carried out for one, six, 12, or 24 hours, and maintained at 20°C (Tables 3 and 4). Also, separate ingredients of the water-softener, sodium hexametaphosphate, sodium tripolyphosphate, sodium chloride, sodium bicarbonate, and sodium carbonate were each tested in concentrations of 5% for 24 hours. The sodium hexametaphosphate was 96% “pure” and the sodium tripolyphosphate was 85% “pure”, as obtained from a chemical supplier.

After each experiment, specimens were recovered on a 75μm sieve and air dried. Shells were mounted on aluminium stubs coated with about 500Å of gold, then examined and photographed with a scanning electron microscope at about 68X and 340X.

| Species                      | Specimens | Age   | Locality       |
|------------------------------|-----------|-------|----------------|
| *Echinocythereis margaritifera* | V, C, J, A* | Modern | Louisiana Shelf, U.S.A. |
| (Brady, 1870)                |           |       |                |
| *Hulingsina tuberculata*     | V, J, A   | Modern | As above       |
| Puri 1958, and *Krithe* *K. producta*, Brady, 1880 | | |
| *Cytherella sp.*             | V, A      | Modern | As above       |
| *Malzella bellegladensis*    | C, J, A   | Pleistocene | Tulane Univ. 201 (see text) |
| (Kontrovitz, 1978)           |           |       |                |
| *Orionina bradyi*            | C, A      | Pleistocene | As above       |
| van den Bold, 1963           |           |       |                |
| *Buntonia morei* (Howe and Pyeatt, 1935), and *Trachy-Grant leberis? montgomeryensis* | V, A | Eocene | Montgomery Landing, Parish, |
| Louisiana (Howe and Chambers, 1935) | | | (see text) |
| *Haplocytheridea montgomeryensis* | V, J | Eocene | As above |
| (Howe and Chambers, 1935)    |           |       |                |

Table 1: Species, ages, and localities of ostracod shells used here.
*V = Valves, C - carapaces, J = juveniles, A = adults.
Table 2: The water used, pH values, and sources.

| Water   | pH   | Source                                                                 |
|---------|------|------------------------------------------------------------------------|
| NLU     | 8.12 | Monroe, Louisiana, tap water, city water system, tap from Bayou Desiard; collected at Northeast Louisiana University. |
| NLU     | 6.85 | As above, but distilled with Stokes Automatic distilled Water Still, Model 171-E. |
| Bossier | 6.59 | Bossier City, Louisiana, tap water, city tap water system, from Red River; collected at Bossier Parish Community College. |
| Shreveport | 7.43 | Shreveport, Louisiana, tap water, city tap water system, from Cross Lake; collected at Wilkinson St. Shreveport. |
| Oasis   | 6.76 | Oasis Water Company, Ft. Worth, Texas distilled, from Ft, Worth city water system. |

Table 3: Concentrations of the phosphatic water softener (Calgon) and duration of trials.

| Water   | Concentration of Calgon (% by weight) | Duration of trials (hours) |
|---------|--------------------------------------|----------------------------|
| NLU tap | 0.0                                  | 3, 6, 12, 24               |
|         | 2.5                                  | 3, 6, 12, 24               |
|         | 5.0                                  | 3, 6, 12, 24               |
|         | 10.0                                 | 3, 6, 12, 24               |
|         | 15.0                                 | 3, 6, 12, 24               |
|         | 20.0                                 | 3, 6, 12, 24               |
| Bossier tap | 0.0                                 | 24                         |
| Shreveport tap | 2.5                        | 24                         |
| Oasis distilled | 5.0                               | 24                         |
|         | 10.0                                 | 24                         |
|         | 15.0                                 | 24                         |
|         | 20.0                                 | 24                         |
| NLU distilled | 0.0                                 | 6, 24                      |
|         | 2.5                                  | 6, 24                      |
|         | 5.0                                  | 6, 24                      |
|         | 10.0                                 | 6, 24                      |
|         | 15.0                                 | 6, 24                      |
|         | 20.0                                 | 6, 24                      |

* 2.5% not used

RESULTS AND DISCUSSION

As expected, at low magnification untreated specimens of living species displayed sharp outlines and distinct features such as ornamentation and pore canals. *Echinocythereis margaritifera* (Brady, 1870), for example, had smooth eye tubercles, distinct marginal denticles, and low rounded spines. At magnifications of X340, spines appeared to be circular in cross-section; shell surface between the spines was nearly smooth with relief of less than three microns. Pore canals had distinct edges, with a few surrounded by papillae, forming an interrupted "protuberant rim" (Sylvester-Bradley and Benson, 1971) (Plates 1 and 2). Fossil ostracods show much variation in condition (Oertli, 1975), but the forms used here, as exemplified by *Haplocytheridea montgomeryensis* (Howe & Chambers, 1935), displayed good preservation, with distinct outlines and pits and pointed marginal denticles.

NLU tap water, alone or with water softener (up to 20%), after one hour, produced no visible damage to modern or fossil shells. Indeed, water alone caused no damage during any trial. Concentrations of 2.5% Calgon and higher, over six hours or more, caused noticeable damage to all modern and fossil shells (Table 4). There was increased deterioration up to a certain concentration, with less than maximum deterioration above that level. We cannot explain this, but it should be noted as it relates to use of phosphates in this manner (Table 4; Pls 1 & 2).

Table 4: Concentrations of phosphatic water softener (Calgon) and duration of trials.

| Water   | Calgon (% by weight) | Duration of trials (hours) |
|---------|----------------------|----------------------------|
| NLU tap | 0.0                  | 3, 6, 12, 24               |
|         | 2.5                  | 3, 6, 12, 24               |
|         | 5.0                  | 3, 6, 12, 24               |
|         | 10.0                 | 3, 6, 12, 24               |
|         | 15.0                 | 3, 6, 12, 24               |
|         | 20.0                 | 3, 6, 12, 24               |
| Bossier tap | 0.0                  | 24                         |
| Shreveport tap | 2.5                | 24                         |
| Oasis distilled | 5.0                | 24                         |
|         | 10.0                 | 24                         |
|         | 15.0                 | 24                         |
|         | 20.0                 | 24                         |
| NLU distilled | 0.0                  | 6, 24                      |
|         | 2.5                  | 6, 24                      |
|         | 5.0                  | 6, 24                      |
|         | 10.0                 | 6, 24                      |
|         | 15.0                 | 6, 24                      |
|         | 20.0                 | 6, 24                      |

* 2.5% not used
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displaying rough, chaotic topography, at 340X. Shells taken from a 20% solution were better preserved than those from 10 and 15% solutions; they were damaged slightly less than those in 5%.

In Oasis distilled with 2.5% Calgon (24 hours) as much as one-half of each shell disappeared. Remaining surfaces were rough, margins irregular, and ornamentation was reduced, while in 5% shells were reduced to irregular fragments with rough surfaces composed of folded organic material. In a 10% solution large holes developed through the shells, margins became ragged, and remaining surfaces were rough. Not even fragments were recovered from 15% solutions, and again, a higher concentration, here 20%, caused less damage than a weaker solution.

There was no evidence that juveniles were more damaged than adults of the same species. There were different degrees of damage among the several species; for NLU tap and NLU distilled water with Calgon, the following modern forms are listed in order of increasing damage: *Echinocythereis margaritifera*, *Hulingsina tuberculata*, *Cytherella* sp., and *Kriitha* sp. cf. *K. producta*.

Some compounds in Calgon (sodium chloride, sodium carbonate, and sodium bicarbonate) used separately in 5% solutions for 24 hours had no effect on the shells. Five percent solutions of sodium hexametaphosphate and sodium tripolyphosphate each caused significant damage during 24 hours and these phosphates appear to be the only ingredients in the water-softener that caused damage.

### CONCLUSIONS

Commonly used water-softener, with sodium hexametaphosphate and sodium tripolyphosphate, in concentrations as low as 2.5% during a six hour period, caused significant damage to ostracod shells. Calgon in different waters caused different degrees of damage, but there was always some alteration.

Alteration could be misinterpreted to be taphonomic, and many surface features resemble those attributed to diagenesis and other post-mortem influences. We are relieved that in taphonomical studies we did not use phosphates for preparation (Kontrovitz, 1987).

The use of phosphatic water-softener, as described, at least would modify some shell features. All taxa are not equally resistant and assemblage composition could be misinterpreted, with possible errors in analyses of diversity, dominance, clusters, and differential transport (Kontrovitz & Nicholich, 1979).

We counsel against the use of phosphatic water-softener or sodium hexametaphosphate or sodium tripolyphosphate to clean ostracod shells or to disaggregate sediment with such shells. If the products are used, we recommend that workers experiment with waters from different sources, use the minimum time possible, and be alert to the possibility of differential dissolution of the taxa involved.

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| Calgon % | Shell damage |
|----------|--------------|
| 0-20%    | None         |
| 2.5%     | Surface features slightly dissolved, denticles worn; surface appeared to have lost flakes (exocuticle?) |
| 5%       | More flaking, exposed lower surface granular; remaining old surface granary (high mag.); solution tracts; 3-5 micron pits. Spines, with septa-like structures. |
| 10%      | Part outer shell lost, newly exposed surface granity. Eye tubercle rough. Enlarged pores. Central scars etched. |
| 15%      | Better preservation than in 2.5, 5, or 10%. High mag., debris strewn, but much of surface between spines smooth. |
| 20%      | Surface much as in 5-10%; some flaking, pores enlarged. |

| Time     | Damage |
|----------|--------|
| 1 hour   |        |
| 6 hours  |        |
| 12 hours |        |
| 24 hours |        |

**Table 4: Damage to ostracod shells in Calgon solutions and times for NLU tap; pH for 0, 2.5, 5, 10, 15, 20% Calgon = 8.12, 7.93, 7.75, 7.49, 7.28, 7.22, respectively.**

**Explanation of Plate 2**

All photographs are of the same specimens of *Echinocythereis margaritifera* (Brady, 1870) shown in Plate 1; in tap water from Northeast Louisiana University, with concentrations of Calgon and times as indicated. Bar Scale = about 60 microns; applicable to all photographs on plate.

- **Fig.1.** Untreated.
- **Fig.2.** Calgon, 0%; 12 hours.
- **Fig.3.** Calgon, 2.5%; 12 hours.
- **Fig.4.** Calgon, 5%; 12 hours.
- **Fig.5.** Calgon, 10%; 12 hours.
- **Fig.6.** Calgon, 15%; 12 hours.
- **Fig.7.** Calgon, 20%; 12 hours.
- **Fig.8.** Calgon, 10%; 24 hours.
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