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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 [$(NaPO_3)_n$] (Jones, 1956) and/or sodium tripolyphosphate ($Na_5P_3O_{10}$). 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 75mμ sieve and air dried. Shells were mounted on aluminium stubs coated with about 500Ä of gold, then examined and photographed with a scanning electron microscope at about 68X and 340X.

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

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

Water	p H	Source
N L U	8.12	Monroe, Louisiana, tap water, city water system, tap from Bayou Desiard; collected at Northeast Louisiana University.
N L U Automatic	6.85	As above, but distilled with Stokes 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 Warer Company, Ft. Worth, Texas distilled, from Ft, Worth city water system.

Table 2: The water used, pH values, and sources.

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, Shreveport tap* Oasis distilled	0.0	24
	2.5	24
	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

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

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).

In NLU distilled water with a 2.5% Calgon, slightly more dissolution took place than in NLU tap. In 5% there was about the same damage as in NLU tap, while in 10% there was a significant loss of "flakes", and holes developed, but there was less damage than in NLU tap, where the outer layer, perhaps the "exocuticle" of Bate and East (1972), was removed. There was a slight increase in dissolution from the 10 to 15% solutions, but less than in NLU tap. In a 20% solution the outer layer was removed; damage was about equal to that in NLU tap with a 5% Calgon.

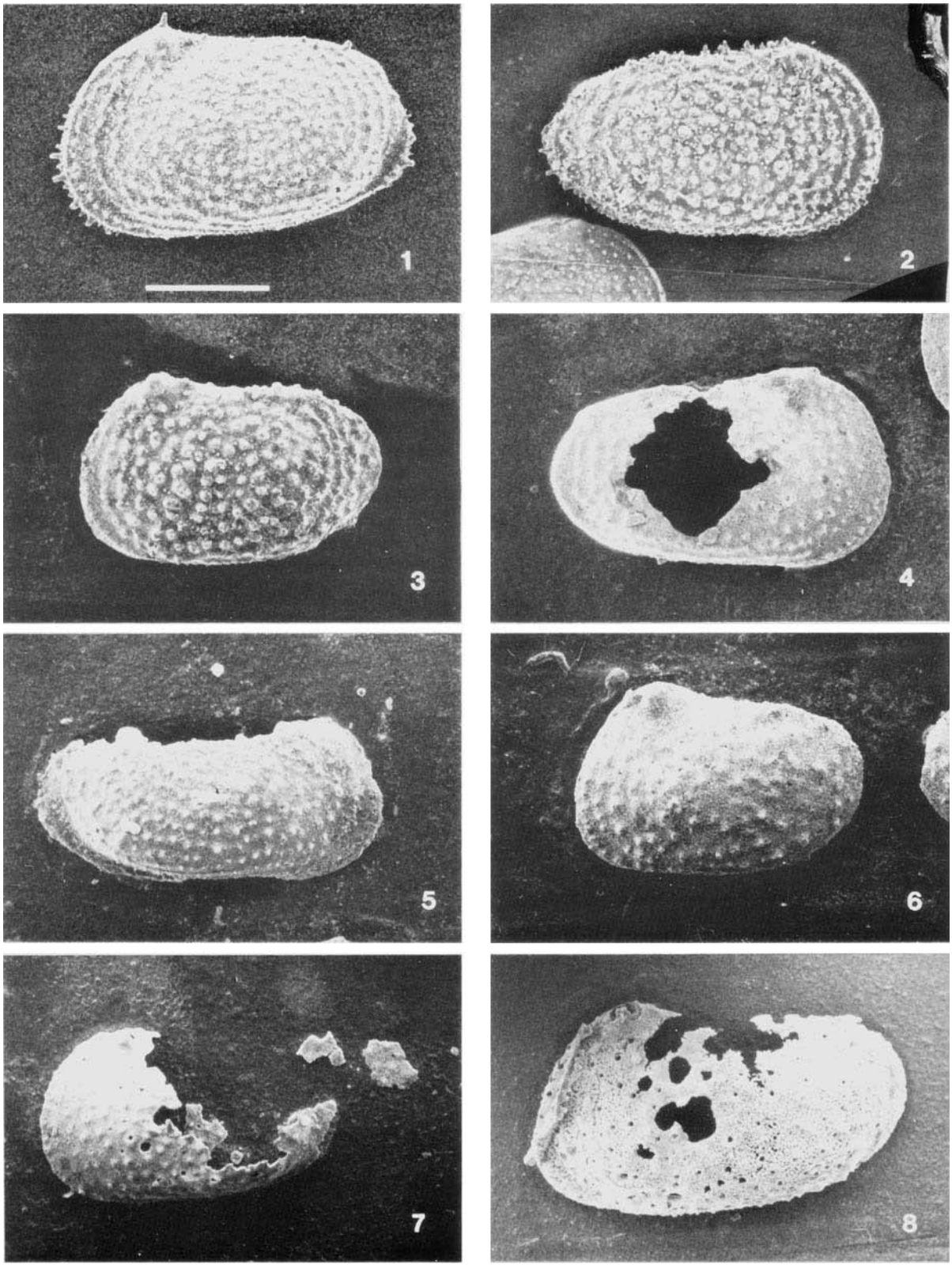
In Shreveport tap with 5% Calgon, only small fragments remained, and although some specimens were identifiable to the generic level, this was only because the taxa were already known to the authors. The damage was less than in NLU tap with 5% Calgon and more than in NLU tap with 10%. In Shreveport tap (10%) only small useless fragments remained; only fragments remained in 15% and 20%, representing thick marginal areas.

In Bossier tap with a 2.5% solution shells were also reduced to fragments. In some places on the fragments a more proximal, somewhat smoother surface (endocuticle?) was exposed. In a 5% solution, again only fragments remained, but these had even rougher surfaces caused by pitting. With 10% and 15% Calgon only small fragments remained,

Explanation of Plate 1

All photographs are of modern *Echinocythereis margaritifera* Brady, 1870) in tap water from Northeast Louisiana University, with concentrations of Calgon and times as indicated. Bar Scale = about 300 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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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 *Krithe* 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 eqully 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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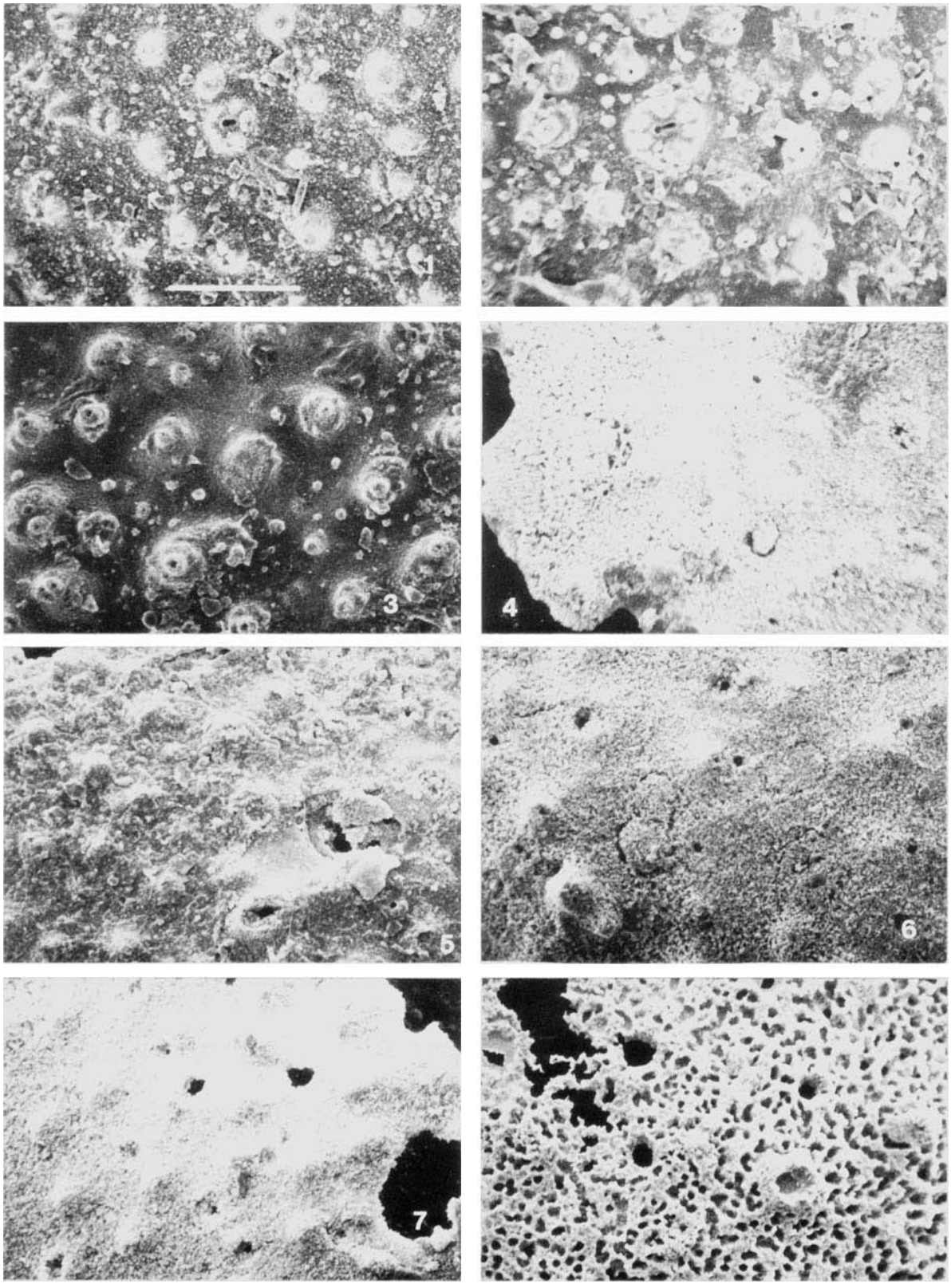
Calgon %	Shell damage
	1 hour
0-20%	None
	6 hours
0%	None
2.5%	Surface features slightly dissolved, denticles worn; surface appeared to have lost flakes (exocuticle?)
5%	More flaking, exposed lower surface granular; remaining old surface grainy (high mag.); solution tracts; 3-5 micron pits. Spines, with septa-like structures.
10%	Part outer shell lost, newly exposed surface grainy. 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.
	12 hours
0%	None
5%	Holes; up to 1/3 shell gone; margins dissolved; marginal denticles gone; eyespot ragged; lateral spines reduced to low knobs; pores enlarged. Pits 2 microns across, larger pits 4-5 microns, porous appearance.
10%	Large portions gone; margins ragged; denticles reduced; normal pores as in 5%; spines now low knobs. High mag., flakey surface; better than 5%.
15%	Outline jagged; eyespot and surface granular, with pits; pores enlarged; Muscle scars etched. Worse than 10%, better than 5%.
20%	Up to 1/2 gone; remainder much altered; pores enlarged; isolated holes; eyespot with rough surface. Better than 5%, worse than 10-15%.
	24 hours
0%	None
5%	One-third to 1/2 shell gone; pores enlarged; denticles, spines reduced; outline jagged; surface granular.
10%	About 1/4 shell missing; margins jagged. Isolated large holes; Spongy appearance, "badlands" topography at high mag. Pits, some through shell.
15%	A few fragments remained, mostly thick areas of hinge, fused duplicature and outer lamella. Pores enlarged; surface coarse, flakey.
20%	Better than 10 and 15%. Equal to 5%. Slightly enlarged pores. Few isolated holes.

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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