The Ancient Shells Were Studied by X-ray Diffraction and Electron Probe

Chen Junhao, Chen Guiqing

Institute of Technology, Jinan University, Guangzhou, China

Email address: c2816@126.com (Chen Junhao), 814713052@qq.com (Chen Guiqing)

To cite this article: Chen Junhao, Chen Guiqing. The Ancient Shells Were Studied by X-ray Diffraction and Electron Probe. Journal of Water Resources and Ocean Science. Vol. 8, No. 6, 2019, pp. 86-93. doi: 10.11648/j.wros.20190806.11

Received: September 27, 2019; Accepted: October 17, 2019; Published: November 5, 2019

Abstract: What changes have they made since the shells were buried in the ground for thousands of years? The geological crystallography book records that the physical properties of the aragonite structure are unstable and will eventually be converted into calcite. This is probably also a theory copied from abroad, and no one in the country has ever studied it. The crystal structure of the test substance can only be measured by an X-ray diffractometer, and geologists seem to not be familiar with crystallography. Now I plan to use X-ray diffraction to verify the correctness of this theory? It turns out that after a long geological time, not only did the aragonite not become calcite, but the grain grew up again - "single crystal" and even calcite decreased, proving that this theory is wrong. Carbon 14 (C^{14}) is used to determine the geological age of some artifacts. Now using the electronic probe to detect the "double Chen weathering hole CC" of the ancient shell, it may be used as another method to determine the geological time.

Keywords: Marine Biology, X-ray Diffraction, Aragonite Structure, Calcite Structure, WDX-Electron Probe, CC-double Chen, Weathered Hole

1. Foreword

What changes will happen to shells after they have been buried in the ground for thousands of years? The geological crystallography textbook [4, 5] records that the physical properties of the meteorite structure are unstable and will eventually turn into calcite. This has become a common sense and can be found on any Internet. If the crystal structure really changes, it will be a long process, the vergeite lattice constant will gradually become distorted, and finally become the calcite structure!

Aragonite gemstones are composed of aragonite (also known as aragonite in crystallography), calcite, iron oxide, and opal. They are secondary minerals and aragonite is one of the main components. In the world, only China's Tibet, Taiwan's Penghu Islands and Italy's Sicily produce aragonite gemstones. According to the above theory, aragonite does not seem to exist in the world, because the aragonite structure contained in these minerals has been transformed into calcite structure after hundreds of millions of years, but in fact they still exist! We have detected that the lattice constant of the aragonite structure gradually transforms into the deformed lattice of the calcite structure at high temperature, but under deep sea high pressure, the Abra profundorum shell and the nautilus pampilium L shell are still aragonite crystals. The aragonite structure is stable under high pressure, which also questions the correctness of this theory. We now use x-ray diffraction to detect the crystal structure of ancient shells, to see if the aragonite or calcite distortion lattice constant can be detected? In order to verify the correctness of the above theory!

The use of electronic probes to detect ancient shells has revealed many weathering holes that vary with the geological age. We call it the “double Chen weathering hole CC”, which may be used as another method to determine the geological time.

2. Materials and Methods

2.1. X-ray Diffraction

In the 1980s, researchers from nanjing institute of paleontology, huang baoyu, provided four kinds of ancient shells: clams with split teeth, clams with split riblets, mussels
with domed shells and mussels with beautiful shells. In 2016, tan yehui, a researcher at the south China sea institute of oceanology, Chinese Academy of Sciences, provided four kinds of ancient shells, including tridacna, tridacna, pipi and clams. The crystal structure was determined by X-ray diffractometer. Aragonite structure conforms to 04-1475 [6]; Calcite structure is consistent with 24-27 [6].

2.2. Electron Microprobe Discuss

Materials as before; The electron probe method was used in the study by south China sea institute of oceanology, Chinese Academy of Sciences.

3. Results and Discussion

3.1. Experimental Results of X-ray Diffraction

In Table 1, the lower bar line such as 0.3400 corresponds to the card 041-1475 [6], which is the aragonite line; the lower dotted line such as 0.3038 corresponds to the card 24-27 [6], which is the calcite line; the lower point, the bar, the point line 0.3336 is the silica line. The line will drift somewhat due to detection conditions, zero calibration, etc. The same is true of the following.

3.1.1. Schistodesmus sp. Prismatic Layer

102 West Point Village, field number ADY201, 1983/9/9. Age: hundreds of years to thousands of years.

All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

3.1.2. Schistodesmus Lampreyanus (Baird & Adams) Prismatic Layer

102, Kenting Village, Shanxi, 1986/12/16. Age: hundreds of years to thousands of years.

All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

3.1.3. Unio Douglasiae (Gray) Prismatic Layer

Field number ADY201, 12/16, 1986/12/16. Years: hundreds to thousands of years.

(i). Unio Douglasiae (Gray) Pearl Layer

Most of the spectral lines conform to the card 041-1475. It's almost all aragonite structure.

The only occurrence of calcite weak line d_{010}=0.3038nm, I/I_{0}=1.64%, containing calcite 0.15% [12]. It's too weak, it's probably stuck, because the stratum corneum does not have it.

(ii). Unio Douglasiae (Gray) Periostracum

All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

3.1.4. Lamprotula sp.

Field number ADY193, 1986/12/16. Age: hundreds of years to thousands of years.

(i). Lamprotula sp. Pearl Layer

All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

(ii). Lamprotula sp. Prismatic Layer

Most of the spectral lines conform to the card 041-1475, it's almost all aragonite structure.

The only occurrence of calcite weak line d_{010}=0.3034nm, I/I_{0}=2.6%, containing calcite 0.23%. It's too weak, it's probably stuck.

3.1.5. Trapezium Liratum (Reeve) Periostracum


Most of the spectral lines conform to the card 041-1475, it's almost all aragonite structure.

There are two strong silica lines d=0.3370nm, I/I_{0}=70.2%; d=0.1824nm, I/I_{0}=55.96%, which is the silica that the ancient shells have adhered to in the soil for many years. Because modern shells never see these lines.

3.1.6. Potamocorbula Laevis (Hinas) Periostracum


All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

3.1.7. Corbicula Fluminea (Müller)


(i). Corbicula Fluminea (Müller) Prismatic Layer

All spectral lines conform to card 041-1475, all are aragonite structures and there are no impurities.

(ii). Corbicula Fluminea (Müller) Periostracum

Most of the spectral lines conform to the card 041-1475, it's almost all aragonite structure.

The strongest spectral line d=0.3336nm is the strongest line of silica SiO_{2} d_{101}=0.33435 nm, and the spectral line d=0.4249
nm is the second strongest spectral line of silica SiO$_2$
d$_{100}$=0.4255 nm. This is the silica that the ancient shells were
buried in the soil for many years and therefore adhered. Modern shells have never seen these spectral lines.

Two weak calcite spectral lines appeared: d$_{104}$=0.3033 nm,
I/I$_0$=8.71% and d$_{113}$=0.2282 nm, I/I$_0$=7.26%, containing
calcite 0.77%. It's too weak, Two weak calcite spectral lines
appeared: d$_{104}$=0.3033 nm, I/I$_0$=8.71% and d$_{113}$=0.2282 nm,
I/I$_0$=7.26%, containing calcite 0.77%. It's too weak, and may
be adhered to.

There is also an impurity spectral line d = 0.3205 nm, I / I$_0$
= 18.38%, I don't know what the substance is, Is also adhesive
impurity.

The ancient Corbicula fluminea shell is the same as the modern Corbicula fluminea shell spectral line.

(iii). Modern Corbicula Fluminea (Müller) Prismatic Layer
Most of the spectral lines conform to the card 041-1475, it's
almost all aragonite structure.

The only one weak impurity line d = 0.3632 nm, I / I$_0$ =
3.21%.

3.1.8. Corbicula Maxima Prime
Unearthed from the water project of Longyan Village,
Shunde, Guangdong. Collector: Zhao Huan Ting and so on.
Measurement date / time 2015-11-27 9:48:41

(i). Corbicula Maxima Prime Pearl Layer
All spectral lines conform to card 041-1475, all are
aragonite structures and there are no impurities.

From the left side of Figure 1. The strongest spectral line d= 0.2875nm absolute strength is equal to I = 4290.7 (this is the
diffraction result of the instrument, omitted in the table.
Generally, the strongest intensity of the spectral line of the
shell is about 700), It is very strong, very narrow, and has less
spectral lines. The conclusion of geological crystallography:
the crystal structure of aragonite is unstable, and it will
eventually become calcite. Now, on the contrary, it will
develop to a higher "aragonite single crystal", indicating the
structure of aragonite, it is stable.

From the X-ray diffraction theory of the crystal Scherrer's
formula [7]: the spectral lines become stronger, narrower and
has few spectral lines. is due the growth and thickening of the
grains in the diffraction direction (preferred orientation
growth), we call it "Single crystalization trend".

The growth process of shell pearl layer is the growth
process of aragonite crystal. Due to the lattice distortion
caused by the vacancy or impurity filling into the crystal
lattice, the crystal face family spacing d changes and the
spectral line becomes wider. New internal stress is always
generated, and the stress is constantly released under the
action of stress. When the shellfish die, the crystal stops
growing and no new internal stress is created. The internal
stress in the shell is gradually released, the vacancy is
gradually filled or the impurities are gradually removed to the
grain boundary to eliminate the internal stress. At this time,
the internal stress is also concentrated on the grain boundary,
so it is easy to be weathered away. Aragonite grain grows
continuously, so; Make spectral lines stronger, narrower and
less!

Because the Corbicula maxima shell is larger, it is less
affected by the surrounding soil, more can reflect the growth
process.

(ii). Corbicula Maxima Prime Prismatic Layer
All spectral lines conform to card 041-1475, all are
aragonite structures and there are no impurities.

The strongest line d =0.2709nm absolute strength is equal
to I = 3406.3, It is very strong, very narrow, and has less
spectral lines. However, because the prismatic layer is
between the pearl layer and the periostracum, the internal
stress is released slowly, so the spectral line is slightly more,
but from its X-ray diffraction pattern, many weak lines have
become almost flattening, the development is similar to the
pearl layer spectra line.

(iii). Corbicula Maxima Prime Periostracum
All spectral lines conform to card 041-1475, all are
aragonite structures and there are no impurities.

From the right side of Figure 1, the absolute intensity of the
strongest line d=0.1980nm is the I=481.16, due to the large
organic matter in the stratum corneum, affecting the single
crysta processl, making absolute The intensity is greatly
reduced and the spectral lines are slightly more

The characteristics of the amorphous diffraction pattern are
as shown in the figure, and the entire scanning angle range (2θ
from 1° -- 2° starts to the several tens of degrees) because the
intensity of the near direct beam is large. And the intensity
rapidly with the increase of the angle to decrease, to a high
angle intensity towards the instrument's background value,
there may be one to several peak values. From the point of
view of Scherrer's formula [7], this phenomenon can be
considered as the grain size is extremely small, and these
crystallites are too small to be affected by external forces, and
the crystal lattice is differently distorted, resulting in the
diffraction peak of the crystal being greatly widened. The
result of overlapping and obscuring.

Incoherent scatter occurs when X-rays are incident on the
sample, and their intensities are also mainly concentrated in
the low angle range, with the result that the background
intensity is increased. There is also background scattering
from the air.
Figure 1. X-ray diffraction pattern of Corbicula maxima: Left picture pearl layer, Middle picture prismatic layer, Right picture periostracum.
Table 1. Shell X-ray diffraction data.

<table>
<thead>
<tr>
<th>Schistodema sp. prismatic layer</th>
<th>Schistodema lampreyanus prismatic layer</th>
<th>Unio douglasiae pearl layer</th>
<th>Unio douglasiae periostracum</th>
</tr>
</thead>
<tbody>
<tr>
<td>d (nm)</td>
<td>I/I (%)</td>
<td>d (nm)</td>
<td>I/I (%)</td>
</tr>
<tr>
<td>Prismatic layer</td>
<td></td>
<td>Unio douglasiae periostracum</td>
<td></td>
</tr>
<tr>
<td>0.3980</td>
<td>2.32</td>
<td>0.3398</td>
<td>75.2</td>
</tr>
<tr>
<td>0.3400</td>
<td>91.4</td>
<td>0.3274</td>
<td>43.6</td>
</tr>
<tr>
<td>0.3275</td>
<td>48.4</td>
<td>0.2702</td>
<td>100</td>
</tr>
<tr>
<td>0.2702</td>
<td>100</td>
<td>0.2484</td>
<td>42.9</td>
</tr>
<tr>
<td>0.2484</td>
<td>52.9</td>
<td>0.2373</td>
<td>46.9</td>
</tr>
<tr>
<td>0.2373</td>
<td>55.4</td>
<td>0.2332</td>
<td>27.9</td>
</tr>
<tr>
<td>0.2335</td>
<td>28.5</td>
<td>0.2105</td>
<td>13.3</td>
</tr>
<tr>
<td>0.2105</td>
<td>22.2</td>
<td>0.1976</td>
<td>39.5</td>
</tr>
<tr>
<td>0.1977</td>
<td>52.9</td>
<td>0.1879</td>
<td>22.6</td>
</tr>
<tr>
<td>0.1879</td>
<td>31.1</td>
<td>0.1814</td>
<td>19.5</td>
</tr>
</tbody>
</table>

Table 2. Lattice constant of shell aragonite structure [8-13].

<table>
<thead>
<tr>
<th>Schistodema sp. shell prismatic layer</th>
<th>Trapezium liratum shell periostracum</th>
</tr>
</thead>
<tbody>
<tr>
<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.500, b=0.805, c=0.576, v=0.232</td>
</tr>
</tbody>
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<table>
<thead>
<tr>
<th>Schistodema lampreyanus shell prismatic layer</th>
<th>Potamocorbula laevis shell periostracum</th>
</tr>
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<tr>
<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.500, b=0.799, c=0.575, v=0.230</td>
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</table>

<table>
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<tr>
<th>Unio douglasiae shell pearl layer</th>
<th>Corbicula fluminea shell prismatic layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.497, b=0.799, c=0.575, v=0.228</td>
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<th>Corbicula fluminea shell</th>
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<tbody>
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<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.494, b=0.795, c=0.573, v=0.225</td>
</tr>
</tbody>
</table>

Lattice constant of ancient shell aragonite structure (nm, nm³)

<table>
<thead>
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<th>Schistodema sp. shell prismatic layer</th>
<th>Trapezium liratum shell periostracum</th>
</tr>
</thead>
<tbody>
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<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
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</tr>
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<tbody>
<tr>
<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.500, b=0.799, c=0.575, v=0.230</td>
</tr>
</tbody>
</table>

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<tbody>
<tr>
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</tr>
</tbody>
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<table>
<thead>
<tr>
<th>Unio douglasiae shell</th>
<th>Corbicula fluminea shell</th>
</tr>
</thead>
<tbody>
<tr>
<td>a=0.497, b=0.797, c=0.574, v=0.227</td>
<td>a=0.494, b=0.795, c=0.573, v=0.225</td>
</tr>
</tbody>
</table>

periostracum

Lamprotula sp. shell prismatic layer

\[ a = 0.497, b = 0.797, c = 0.574, v = 0.227 \]

Pinctada margaritifera shell periostracum

\[ a = 0.497, b = 0.797, c = 0.574, v = 0.227 \]

Lamprotula sp. Shell; Modern periostracum

\[ a = 0.496, b = 0.796, c = 0.574, v = 0.227 \]

**Table 3. Calcite content of ancient and modern shells [8-13].**

<table>
<thead>
<tr>
<th>Calcite content of ancient shells: Well below 1%, it's almost nonexistent</th>
<th>Lamprotula sp. Shell prismatic layer</th>
<th>Corbicula fluminea shell periostracum</th>
<th>0.77%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Schistodesmus sp. Shell prismatic layer</td>
<td>0%</td>
<td>0.23%</td>
<td></td>
</tr>
<tr>
<td>Schistodesmus lamproyanus shell prismatic layer</td>
<td>0%</td>
<td>0%</td>
<td></td>
</tr>
<tr>
<td>Unio douglasia shell prismatic layer</td>
<td>0.15%</td>
<td>0%</td>
<td></td>
</tr>
<tr>
<td>Unio douglasia shell periostracum</td>
<td>0%</td>
<td>0%</td>
<td></td>
</tr>
<tr>
<td>Lamprotula sp. shell pearl layer</td>
<td>0%</td>
<td>0%</td>
<td></td>
</tr>
<tr>
<td>Calcite content of modern shells</td>
<td>Pinctada chemnitzi shell pearl layer</td>
<td>Corbicula margaritifera shell periostracum</td>
<td>8.31%</td>
</tr>
<tr>
<td>Pinctada chemnitzi shell periostracum</td>
<td>100%</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>Ostrea rivularis shell Most of the majority</td>
<td>100%</td>
<td>100%</td>
<td></td>
</tr>
<tr>
<td>Mytilus edulis shell</td>
<td>15.19%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chama dunker shell</td>
<td>20.03%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Abra profundorum shell</td>
<td>14%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Modern Corbicula fluminea shell prismatic layer</td>
<td>29.92%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

All prismatic layers of pearl shells contain less than 1% of calcite spectral lines.

### 3.2. Electron Microprobe Results

Elements with atomic Numbers below 11 (Na) cannot be measured, so; CaCO\(_3\) can only be calculated from CaO molecular formula.

\[
\text{CaO} + \text{CO}_2 \rightarrow \text{CaCO}_3 \\
56.0794 + 100.0892 = 156.1686
\]

M\(_{\text{CaCO}_3}\) = (100.089 \times 56.0794)\times M_{\text{CaO}} = 1.78478 \times M_{\text{CaO}}

For ancient *Schistodesmus* sp. Shell:

Inner layer: M\(_{\text{CaCO}_3}\) = 1.78478 \times 48.165 = 85.9639;

Surface layer: M\(_{\text{CaCO}_3}\) = 1.78478 \times 45.125 = 80.5377.

The same is true of the following.

In the table summation \( \Sigma \) is equal to the summation calcium carbonate and the rest of the oxide. Modern shells of summation measurement results of electron probe \( \Sigma \) equal to about 100%, not necessarily is 100, this is due to the error due to certain factors, normalized to 100%, but the ancient sea shells \( \Sigma \) and 100% have a certain gap, this is the result of ancient shell one thousand weathering, we define: \( CC = 100 - \Sigma \) for "double chen weathering hole", all the parts of the CC in the shell is not the same, expressed by average. According to our test scores of modern shells, general results: \( \Sigma = 98.5, \) so imagine CC value within 5 negligible, as 0, namely "Double Chen weathering hole" is 0. Because the scope of the electron probe test is very small, the important data need to be measured and averaged.

Will ancient *Schistodesmus* sp. Shell; Ancient *Schistodesmus lamproyanus* shell; Ancient *Unio douglasia* shell; Ancient *Lamprotula* sp. Shell; Modern *Pinctada fucata*
4. Conclusions

1. We studied the X-ray diffraction results of 16 samples of 8 ancient shells and modern shells as shown in Table 1. Most of them are aragonite structures of calcium carbonate, and the crystal grains are relatively large, a small amount of calcite structure. Only the stratum corneum of the striatum and the stratum corneum of the river scorpion are with silica and a little impurity. The crystal structure, lattice constant and unit cell volume of ancient shells, it are not different from those of modern shells [8-13] Crystallography books and textbooks the conclusion record that the physical properties of the aragonite structure are unstable, after many years be converted into a calcite structure is incorrect. Moreover, China’s Tibet, the Penghu Islands in Taiwan Province, and the Sicily in Italy produce aragonite gemstones (mainly aragonite structures) that have survived for hundreds of millions of years.

2. From the Corbicula maxima Prime shell pearl layer and prism layer buried in the ground for thousands of years, the aragonite structure has developed to a higher degree of crystallization - "single crystal", making the spectral line stronger, narrower and less ! It is even more impurity-free than modern shells, which means that the aragonite structure grains are grow and more stable. Further reversing the conclusion that geological crystallography is unstable about the crystal structure of aragonite and will eventually become calcite. Because there are very few calcite lines, maybe even calcite turns into a aragonite!

3. many modern shells has calcite structure as shown in the table 2, but the ancient shells basically have no calcite structure, It shows that after a long period of time, the calcite of the shell became aragonite.

4. The "shellfish science program" considers that the shellfish shell prism layer is a calcite structure [9-13] is wrong. It is confirmed from the above shells and ancient shells that the prism layer of the shell is all aragonite structure.

5. We studied the electron probe detection of 8 kinds of ancient shells and modern shell samples, and found

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>Fe₂O₃</th>
<th>MnO</th>
<th>SiO₂</th>
<th>MgO</th>
<th>TiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>CaCO₃</th>
<th>SumΣ</th>
<th>CC</th>
<th>CC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ancient Schistodesmus sp.</td>
<td>0.02</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Inner layer</td>
<td>0.14</td>
<td>0.01</td>
<td>0.02</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Surface layer</td>
<td>0.19</td>
<td>0.01</td>
<td>0.03</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Modern Tegillarca granosa</td>
<td>0.01</td>
<td>0.00</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Shell sample thicker, better test results!

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**Table 5. Comparison of the “double Chen weathered hole CC” result between Ancient and modern shells.**

<table>
<thead>
<tr>
<th>Ancient shell species</th>
<th>Oxide summation</th>
<th>Double Chen weathered hole</th>
<th>CC</th>
<th>Moder shell species</th>
<th>Oxide summation</th>
<th>Double Chen weathered hole</th>
<th>CC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Schistodesmus sp.</td>
<td>84.09</td>
<td>15.9</td>
<td></td>
<td>Pinctada martensi shell</td>
<td>100.00</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Schistodesmus lampreyanus</td>
<td>80.08</td>
<td>19.9</td>
<td></td>
<td>Lamprotula mansuyi shell</td>
<td>100.30</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Unio douglasiae</td>
<td>80.73</td>
<td>19.3</td>
<td></td>
<td>Mactra antiquate shell</td>
<td>100.00</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Lamprotula sp.</td>
<td>87.16</td>
<td>12.8</td>
<td></td>
<td>Arca (Anadara) granosa shell</td>
<td>100.00</td>
<td>0</td>
<td></td>
</tr>
</tbody>
</table>
many voids - "Double Chen weathering hole CC" is shown in Table 5! We hope that it will become another way to apply ancient shells and even ancient artifacts to detect the age!

References


