RESEARCH NOTE

A new technique for retrieving calcareous microfossils from lithified lime deposits

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ABSTRACT: Digestion in cold, highly concentrated acetic acid, a technique commonly adopted in conodont studies, is successful in retrieving calcareous microfossils, such as planktic and benthic foraminifera as well as ostracods, from strongly lithified lime deposits (calcilutites, marly calcilutites and fine-grained calcarenites). Moreover, in cases where the standard disaggregation techniques employing hydrogen peroxide or Desogen do not produce clean specimens, acetic acid treatment of the residue will remove residual encrustations and will also improve liberation of the small-sized fraction.

INTRODUCTION

One of the major problems for micropaleontologists is the liberation of clean and undamaged microfossils from strongly lithified matrix. Normally, lithified samples require long disaggregation time to eliminate adhering matrix from the specimens, often with very poor results.

The aim of this note is to describe a new and surprisingly effective technique to disaggregate highly lithified rocks in a short time. I have applied this treatment to Tertiary sediments of the central and southern Apennines (Italy).

Samples of lithified lime deposits (calcilutites, marly calcilutites and fine-grained calcarenites) are treated with a solution of acetic acid for retrieving calcareous microfossils such as planktic and benthic foraminifera, as well as ostracods. The solution is called ethanoic acid CH_3COOH, made up of 80% acetic acid and 20% H_2O. The concentration of the acid is close to the undiluted glacial acetic acid. The acetic acid causes a very slow reaction that disaggregates the rocks without destroying and corroding fossil content.

In 1962, Bourdon first proposed the use of acetic acid for retrieving microfossils from very hard lime lithologies. The method, known as "hot acetolyse", was subsequently modified by Lethiers and Crasquin-Soleau (1988) who reduced the disaggregation time that however lasted some days.

The technique proposed here, based on cold-disaggregation with acetic acid, resembles the technique adopted for conodont studies (Graves and Ellison 1941, Stone 1987, Stouge and Boyce 1983). In particular, it resembles the technique adopted by Stouge and Boyce 1983, who used the same acid but with different dilution (they used a solution made up of 10-15% acetic acid and 85-90% H_2O). Text-figure 1 shows the differences between the technique here proposed and that of Stouge and Boyce 1983.

LABORATORY PROCEDURE

The samples are broken down into small fragments of about 5 mm in diameter, placed in a beaker and covered with acetic acid. The small size of the fragments is recommended to obtain a better result. The level of acetic acid must be 2 cm higher than

Stouge and Boyce 1983	This work
1) dilution of acetic acid made up of 10-15% of CH_3COOH and 90-85% H_2O	1) dilution of acetic acid made up of 80% of CH_3COOH and 20% H_2O
2) disaggregation time at least 1 week to obtain the first results	2) disaggregation time at least 2 hours
3) change of acid at least 2-3 times	3) no change of acid
4) to keep the sample in a plastic or polyethylene bucket	4) to keep the sample in a beaker to continuously check the disaggregation
5) no ultrasonic cleaner	5) use of ultrasonic cleaner
6) size of fragments about 5 cm in diameter	6) size of fragments about 5 mm in diameter

TEXT-FIGURE 1
Comparison between the Stouge and Boyce 1983 disaggregation technique and the technique of this work.

Lithologies	Time/hour Acetic acid	Time/hour ultrasonic cleaner
fine-grained calcarenite	at least 10	at least 2-3
calcilutite	5-7	1-2
marly-limestone	5	1-2
marl	2-3	1-2
shaly-marl	2	1-2

TEXT-FIGURE 2
Times of dipping in acetic acid and of the ultrasonic cleaner treatment for different studied lithologies.

the fragment level. As the disaggregation procedes slowly, it is possible to continuously check the deposition of the fine residue on the bottom of the beaker and the swelling of the biggest rock fragments. The product of this vigorous but slow action of the acetic acid is the breaking of the links between particles.

In cases where the amount of acetic acid used to cover the rock fragments is not sufficient, the sample absorbs all the acetic acid and hardens. In such cases it is sufficient to place the sample into an ultrasonic cleaner to dissolve it.

The time of disaggregation varies with the kind of rocks; 6-7 hours are sufficient for calcilutites and marly calcilutites, while at least 10 hours are necessary for fine-grained calcarenites (text-fig. 2).

The porosity and lithification degree of the rocks may influence the times plotted in text-figure 2 and therefore, I suggest to check the indicated times and, if necessary, modify them.

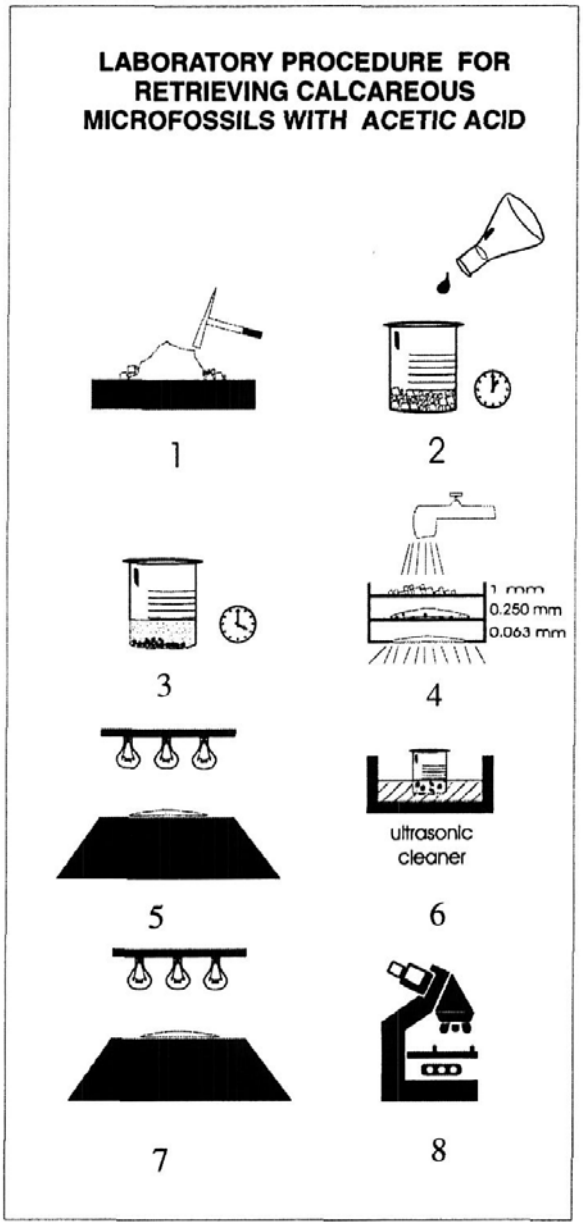
The disaggregated samples must be washed in abundant water, sieved through the standard sieves according to ASTM (18, 25, 150, and 230 Mesh) and dried. Be careful of the strong effervescence caused by the contact of the acid with the water and avoid contact between skin and washing residue.

After this procedure, the residue should be completely free of inorganic matter and the foraminifera should be completely clean, otherwise to remove the residual encrustations and clay material the residue is dipped again in a beaker containing water diluted Desogen and placed into an ultrasonic cleaner for one to two hours. Experiments in the use of ultrasonic vibration for long time intervals have proved to be successful in mechanically cleaning individual specimens without breaking them. In particular the fine-grained deposits produce a clean microfossil assemblagehe. The times are plotted in text-figure 2.

Text-figure 3 shows the main steps of this cold-disaggregation with the acetic acid for retrieving calcareous microfossils.

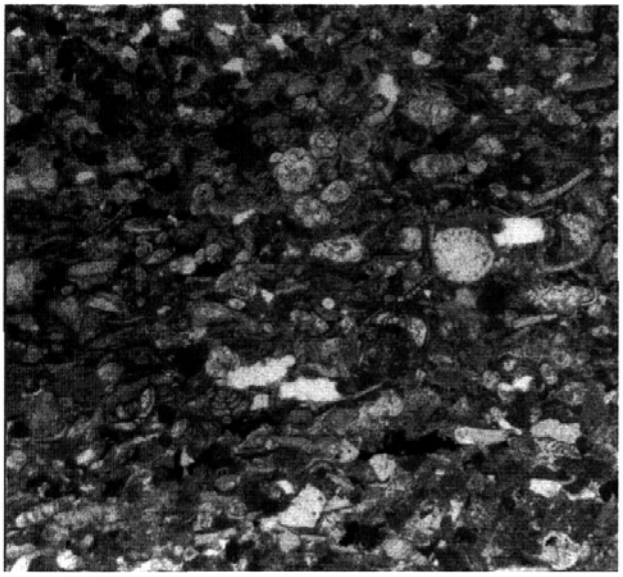
Plates 1 and 2 (figs. 1, 2, 3) show the microfacies of three different rocks samples and the washed residues after the acetic acid treatment; Plate 2 (fig. 4) shows the quality of preservation of foraminifera after the treatment with hydrogen peroxide (4a) and with acetic acid (4b) (text-figure 4).

Warning: It is very important not to modify the percentages of the acetic acid because the addition of water, as well as the exposure of the beaker with the sample to high temperature, leads to an uncontrollable reaction that can destroy the organic content.



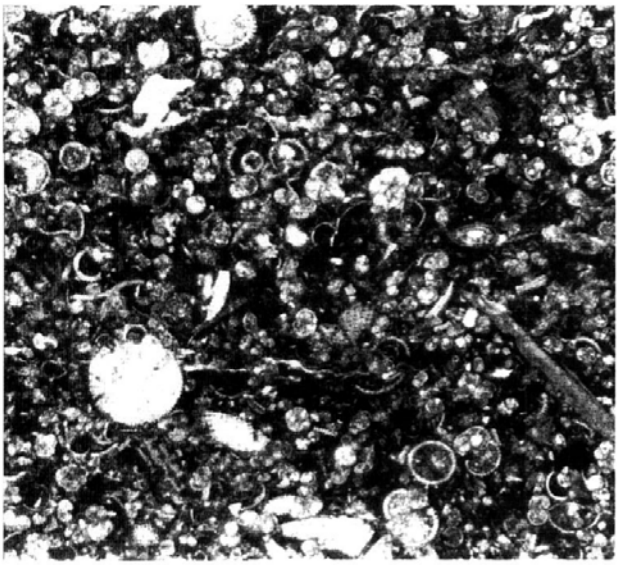
TEXT-FIGURE 3
Laboratory procedure.

1) Break the sample into small-size fragments;
2) Cover the broken down sample with acetic acid;
3) Wait for the residue disaggregation according to the times plotted in text-figure 2;
4) Wash the sample with abundant water and sieve;
5) Dry the residue;
6) Place the residue, with water diluted Desogen, in the ultrasonic cleaner according to the times plotted in text-figure 2;
7) Dry the residue;
8) Residue is ready for the micropaleontologic analysis.

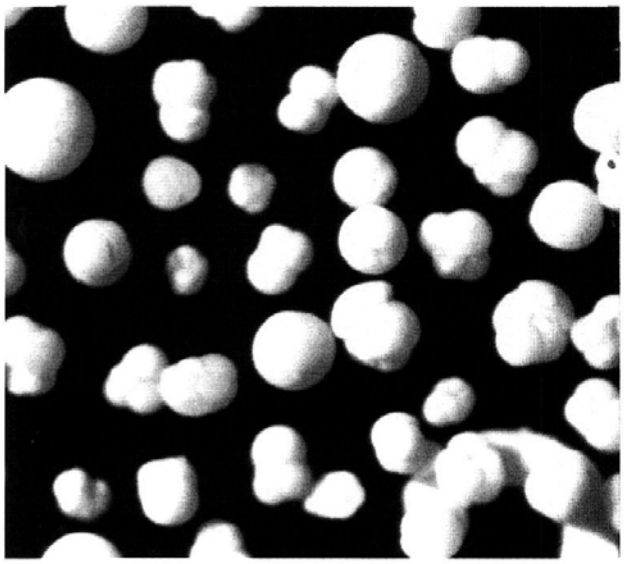


1a

1b



2a



2b

PLATE 1

1a Microfacies of a fine-grained calcarenite (Queglia Unit, central Apennines, Italy), ×200

1b Washed residue obtained from a fine-grained calcarenite (Queglia Unit, central Apennines, Italy), ×200

2a Microfacies of a calcilutite (Chert Calcilutite Member, Bolognano Fm., central Apennines, Italy), ×200

2b Washed residue obtained from a calcilutite (Chert Calcilutite Member, Bolognano Fm., central Apennines, Italy), ×200

1a	Fine-grained calcarenite (Queglia unit)	1b	Washing residue (10 hours dipped in acetic acid)
2a	Calcilutite (Calcilutiti with chert Member, Bolognano Fm.)	2b	Washing residue (6 hours dipped in acetic acid)
3a	Marly-limestone (Orbulina Limestones Member, Bolognano Fm.)	3b	Washing residue (4 hours dipped in acetic acid)
4a	Specimens preservation of hydrogen peroxide treated Pietra Leccese sample	4b	Specimens preservation of acetic acid treated Pietra Leccese sample (30 minutes dipped in acetic acid)

TEXT-FIGURE 4
Kind of disaggregated rocks and their relative times of immersion in acetic acid.

Moreover, when the standard technique (hydrogen peroxide or Desogen) does not produce a complete disaggregation of the samples, and therefore the matrix adheres to the specimen tests or remains inside the tests, they can be further treated using this technique to remove residual encrustations. In these cases the washed residues have to remain in the acetic acid less than one hour, but the reaction must be constantly checked.

The reaction mechanism of the acetic acid is not yet very clear but it is possible to observe that this cold reaction permits the acetic acid to penetrate slowly into the rock fragments and destroy the links between the particles as testified by the swelling of the fragments. This observation disagrees with that of Lethiers and Crasquin-Soleau (1988) who suggest a corrosion reaction providing a volume decrease of the rock fragments.

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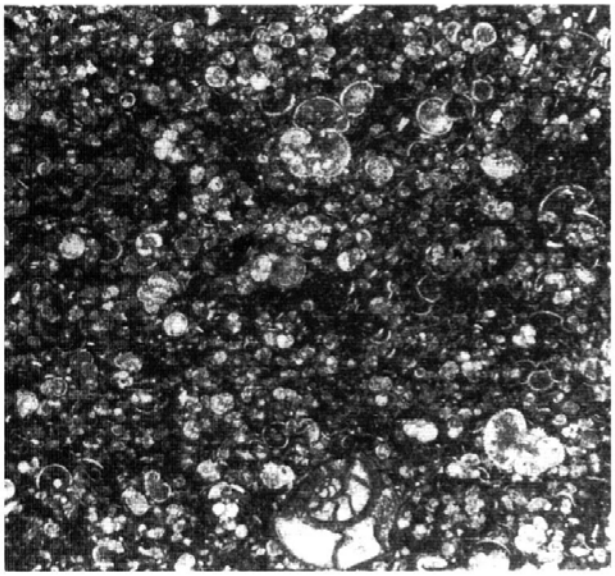
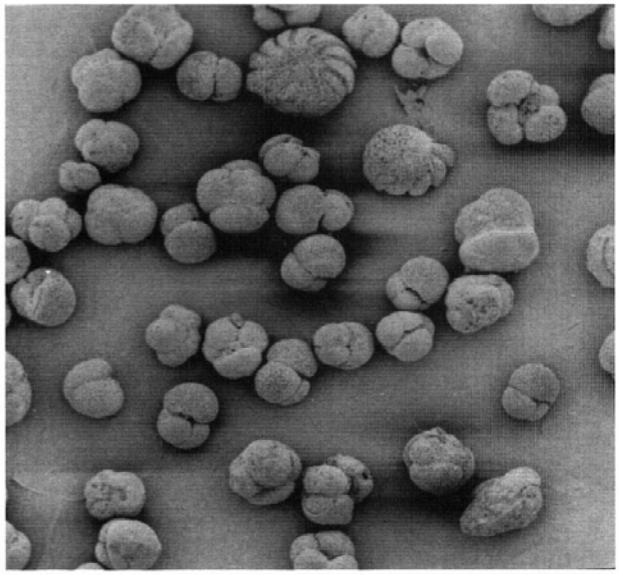


3a



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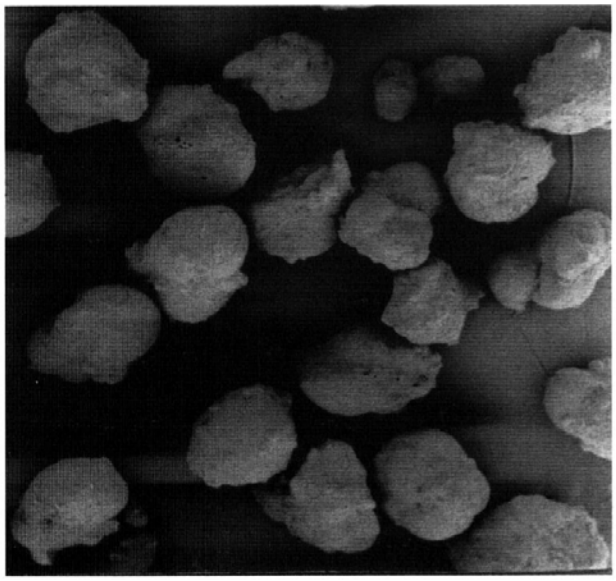
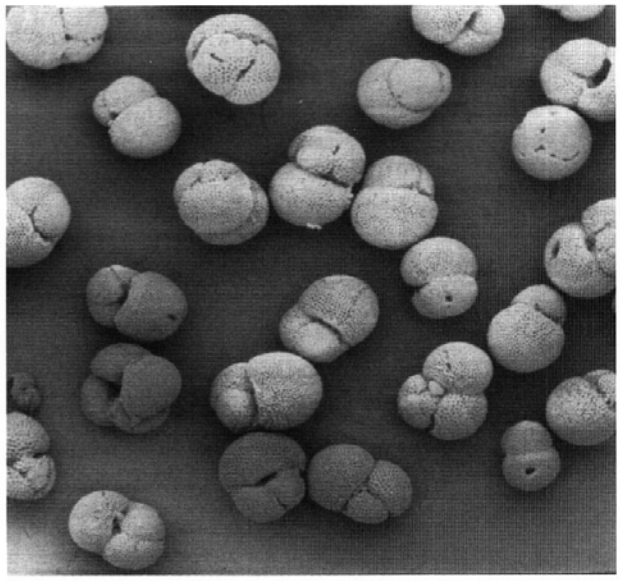


4a



4b

PLATE 2

3a Microfacies of a marly-limestone (Chert Calcilutite Member, Bolognano Fm., central Apennines, Italy), ×200;

3b Washed residue obtained from a marly-limestone (Chert Calcilutite Member, Bolognano Fm., central Apennines, Italy), ×200;

4a Specimens preservation of hydrogen peroxide treated Pietra Leccese sample (south Apennines, Italy), ×200;

4b Specimens preservation of acetic acid treated Pietra Leccese sample (south Apennines, Italy), ×200;