CAUSES OF UNRELIABILITY IN MICROFOSSIL SAMPLES

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Summary

Microfossil assemblages are not necessarily contemporaneous with the sediment from which they were collected; anomalous material can easily be present. This can be a result of natural contamination by processes such as recycling and stratigraphic leakage, which lead to a mixing of fossils from different horizons. Human errors in collecting, labelling and processing of fossil material can also result in contamination and unreliability of samples.

The possible causes of sample unreliability are discussed and ways of recognizing (and, where possible, avoiding) contamination are outlined.

Introduction

Accurate and meaningful interpretation of microfossil samples requires the assessment of various possible sources of error. The interpretation and evaluation of an assemblage based solely on its contents can be as accurate as is humanly possible, yet can give erroneous results and conclusions.

Various factors can make a microfossil assemblage unrepresentative of the horizon from which it is supposed to come, thus making the conclusions obtained from the sample unreliable. These factors include contamination in the laboratory, mixing of assemblages in nature, and misplacing, misidentification or contamination of samples as a result of human error.

This paper is based upon research by the authors on several different groups of microfossils.

Collecting of samples

Samples for micropalaeontological investigation can be collected by one of two methods; by hand, or by mechanical means.

Hand Collected Samples:-

Samples collected by hand are usually collected from natural exposures such as streams and cliff-faces and such artificial exposures as excavations and road-cuttings.

Before undertaking systematic collecting in the field, the collector is best advised to study the succession thoroughly, so that he knows exactly from which horizon/zone etc. each sample came. The help of a local geologist as guide is especially advantageous when dealing with unfamiliar successions, particularly in foreign countries. Far too many cases have occurred of material being collected from either above or below the horizon from which the collector *thought* he collected it. Mistakes such as this can have serious repercussions by producing a false palaeostratigraphical picture.

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Often a certain element of statistical bias is introduced by collecting from the more obvious or easily accessible horizons rather than collecting uniformly. To guard against this, either fixed-interval sampling or random sampling should be undertaken. For detailed stratigraphic work, fixed-interval sampling is advisable, samples being collected at unit distances up the succession. This however is often impossible, because of changing lithology, lack of exposure, or sheer inaccessibility of outcrop. (For details of sampling techniques see, Cochran, 1963; Krumbein, 1965).

In certain areas, weathering diagenesis and metamorphism can present problems, due to partial or complete destruction of the fossils. Such processes include leaching, changes in the oxidation-reduction potential (Eh), changes in the hydrogen iron concentration (pH), and post-diagenetic alterations brought about principally by pressure, temperature and the introduction of new chemical substances (Tschudy, 1966).

The extent of weathering is controlled to some degree by lithology. Hard siliceous shales, limestones, and cherts are much more resistant than soft shales, clays and unconsolidated sediments. Sediments directly underlying bentonite beds are usually silicified and much more resistant to chemical and physical changes and are thus often a good source of organic-walled microfossils.

Removal of weathered and decayed surfaces of rocks is therefore an essential first step, prior to collecting samples. This removal of the surface layers also helps to avoid contamination by extraneous micro-organisms, e.g. modern pollen and spores which may adhere to the surface. Samples for palynological study must always have the surface layers removed or, if sufficiently consolidated, may be scrubbed and washed thoroughly with distilled water (Gray, 1965; Hay 1965; Wilson, 1971).

Where it is necessary to collect samples from beneath an overburden of soil or talus, a spade can be utilized to remove the overburden and expose the sediments. A screw auger can be utilized here, and also for collecting samples of unconsolidated sediments, although some mixing of material from different horizons always occurs.

Generally, unconsolidated sediments require special collecting techniques. These are best collected by forcing a clean plastic tube into the sediment, closing of the upper end of the tube then creates enough suction to retain a core of sediment when the tube is withdrawn. Again however, as with a screw auger, some mixing of material from different horizons can easily occur. (Faegri, and Iversen, 1965).

Mechanically collected samples:-

The most commonly encountered mechanically collected samples are termed 'well samples', these being retrieved in the drilling of oil wells. Samples from present day wells are usually of three types; conventional cores, sidewall cores and cutting samples. Cable-tool drilling is becoming increasingly rare, but is still being used for shallow oil wells in some areas and for water-wells.

Conventional cores are cylinders of the rock penetrated by the bit; taken by coring devices which replace the drilling bit when a core is required.

Sidewall cores are samples taken after a well has been drilled. A device with a number of hollow cylinders is lowered down the well to the desired depth; explosive charges then shoot the cylinders into the sides of the hole. Cable is used to connect the cylinders to the main coring device so that they can be retrived together with a plug of the sidewall.

Cutting samples are fragments of the rock produced by the rotary drill bit cutting its way through the rock. These fragments are brought to the surface by the circulatory fluid (either mud or water) used during drilling operations.

Conventional cores are the most reliable of these three types of samples. Even these however can be misleading, since fragments of rock can be knocked loose from upper

sediments during the lowering of the coring device. In cores of a porous rock such as sandstones, a further source of contamination is that acid-insoluble microfossils may be forced in by pressure of the drilling fluid.

The main problem with sidewall cores is that a certain amount of drilling mud which cakes on the sides of the hole, may be retrieved. This mud may well contain microfossils from other levels, so anything which looks like caked mud should be carefully removed.

The most unreliable type of well sample, and unfortunately the most common, are the cutting samples. Exact depths of these are not easily known owing to the time-lag required for them to be circulated to the surface. Such samples are also susceptible to contamination due to cavings, lost-circulation (drilling fluid entering rock cavities) and additives in the drilling fluid. (Traverse *et. al.* 1961).

If wells are drilled with a gas rather than water or mud then more reliable samples can be obtained. Circulation of material to the surface is much quicker and caving is less of a problem as no swelling etc. (often due to wetting) of poorly consolidated sediments takes place.

Another type of sample is obtained from a shell and auger boring. This is commonly employed in site investigations in this country on poorly consolidated sediments, e.g. London Clay etc. This involves the repeated dropping of a steel cylinder which cuts its way through the sediment leaving a relatively undisturbed core. Undisturbed samples obtained for testing for engineering purposes are usually taken in 4" diameter thin walled steel sampling tubes, 18" long, sealed with wax and screw caps (commonly referred to as a U4). These usually provide reliable samples. Since the level of the sample is accurately known and these borings are usually cased throughout, thus eliminating the danger of caving. The outer layer of extruded samples should always be discarded as the steel tubes are reused for different jobs and they are not always thoroughly cleaned. Mr. C. King, of Paleoservices Ltd., found in one such sample from the London Clay, well preserved Kimmeridgian ostracods and foraminifera from a previous site investigation.

Labelling of Samples in the field:

To minimise the risk of error, all samples should be numbered and labelled accurately whilst in the field; duplication of information in a notebook is advisable. Information such as locality, horizon (or measured distance/depth from a datum line), date and collector should be recorded and not just left to memory. Mislabelled or unlabelled samples, often a consequence of trusting to memory, can lead to completely erroneous conclusions and as often as not represent a waste of collecting time. The importance of accurate labelling cannot be overemphasised.

Sources of Contamination

Transport of samples:

Hard consolidated samples require the least care in packing and transport, provided that they are adequately labelled. If contamination during transport is suspected, they can easily be washed if they are required for palynological study.

Finely divided, friable and unconsolidated sediments however, require a great deal more care. Ideally, such samples should be thoroughly dried before packing and placed in leak-proof bags. At all costs loosely tied bags containing unconsolidated or friable material should not be placed side by side during transport; as spillage, from one to another can easily occur.

Bags of loosely woven material should not be used as they may allow dust, bearing contaminants, to pass through. Even finely woven bags have disadvantages, as dust can collect in the seams and corners. Ideally, each bag should be discarded after one use.

Strong polythene bags with well sealed joints are ideal for certain samples, although if used for palynological samples they can, when warm and containing moist material, act as

incubators for bacteria and fungi which readily attack and destroy organic-walled fossils, and, also, destroy or render illegible labels placed inside the bag.

Airborne Contaminants:

Airborne contaminants are most commonly of modern spore and pollen material. Contamination of this sort therefore only concerns the palynologist.

Obviously the ease with which modern contaminants are recognised depends upon the age of the spore-pollen material with which one is working. If the material is Recent or even near-Recent then the problem of differentiating modern contaminants is far more acute than when dealing with much older assemblages.

Ideally a palynologist should be familiar with what is likely to turn up in the air from the local flora. Greased plates are put out by some palynologists; these are examined at intervals to see what is in the air at the time.

When working with more ancient fossil spores and pollen, the problem of differentiating modern material is not very great. Modern spores and pollen differ from recent forms in body colour, sheen, and reaction to staining and are thus readily identified. Older sediments are nearly always compacted and, in the process, organic walled fossils are compressed, whilst modern material shows no compression.

Occasionally palynological samples may be contaminated in the laboratory by dust from other samples, particularly when samples are being crushed prior to chemical processing. This is a somewhat more serious problem and is much more difficult to resolve. To allow for such an incident occuring, it is advisable to keep abreast on what materials are being processed by colleagues in the laboratory. Palynological samples, for this reason, are best kept during the course of chemical treatment in covered beakers in a fume-cupboard with an air updraught continually switched on.

Calcareous nannoplankton present a problem as contamination due to airborne dust is very difficult to detect. Since they are very small (1 - 10μ being the norm) airborne chalk dust in the laboratory can contain many individuals in suspension (Echols and Levin, 1964). This dust can be trapped in crevices in the skin of the hands and under finger nails or even adhere to the surface of the skin itself. If more than one sample is being handled, then care must be taken to keep the hands clean by thorough washing (Hay, 1965).

Atmospheric contamination must have occurred throughout geological time, but this is a problem which is impossible to resolve.

Contamination in the Laboratory:

The mains water supply to the laboratory, particularly if it has been stored in an open reservoir, is seldom free from micro-organisms: it often contains modern pollen and spores, dinoflagellates, desmids and diatoms and occasionally even fossil material. Such water is an obvious source of contaminants if used in the processing of palynological samples. To guard against this, distilled water should *constantly* be used in the palynological laboratory. Further more, all apparatus should be thoroughly cleaned out and rinsed in distilled water before use.

(a) *Palynomorphs.* Mortars and pestles used for crushing consolidated samples must be carefully scrubbed clean before and after use. Pitting of the pestle can easily result in material being trapped in the crevices, so ideally a damaged pestle should be discarded. A partial answer to contamination during crushing is to line the pestle with several layers of aluminium foil on heavy duty polythene, discarding and replacing the lining after each sample is crushed.

Glassware is a major source of contamination as palynomorphs tend to adhere to glass surfaces when wet. This is particularly true of pipettes whose small diameter makes cleaning of them difficult and it is perhaps advisable to use pipettes which can be discarded after one use (Wilson, 1971).

All glassware, including sinter funnels, used during the processing of organic-walled microplankton should be cleaned with a strong oxidizing acid, such as nitric acid or concentrated chromic acid. Ideally, glassware should be kept immersed in a bath of acid until needed. For calcareous nannoplankton hydrochloric acid should be utilized for cleansing purposes.

A further contamination risk occurs when commercial acids are used during processing. Sediments at the bottom of acid containers often contain spore and pollen material. To minimise this risk, it is preferable to use analar reagents which have a higher purity but, unfortunately, these are much more expensive than commercial acids. Alternatively, if this is impossible, acids should be poured carefully, taking care not to disturb the sediment, and the lees of the acid should be discarded.

(b) *Larger microfossils.* Sieves used in conjunction with the larger microfossils such as foraminifera often retain material trapped in the mesh which can contaminate other samples.

Two main types of sieve are commonly used, wet sieves and dry sieves. These are best cleaned by scrubbing thoroughly with a wire brush under running water.

When using wet sieves for concentrating calcareous foraminifera, a useful method (A.D.K.) is to immerse the sieve in a solution of methylene blue after each sample is sieved. This stains any adherent calcareous material blue so that residual contaminants can easily be distinquished from the contents of a later sample.

An ultrasonic cleansing tank, together with a non ionic detergent, should be utilized to clean all kinds of apparatus from microscope slides to sinter funnels. These are immersed in a water filled tank where electrically generated ultrasonic vibratory waves dislodge any adhering material.

(c) *Silicoflagellates.* These have been found as contaminants in Palaeozoic sediments from Norway (Sarjeant *pers. comm.*). The silicoflagellates are typically Tertiary forms so their occurrence, as the only microfossils in a Palaeozoic sediment warranted investigation. It was discovered that the sample was contaminated by an abrasive powder used in the laboratory as a cleaning and scouring agent. Whilst processing siliceous microfossils such as silicoflaggellates, abrasive powder containing finely divided silica should be carefully avoided.

Sedimentary Contamination

Reworking

Reworking is an integral part of sedimentation, involving the redeposition of older fossils into younger sediments. Problems caused by this are difficult and can occur in any type of sediment regardless of collecting techniques and scare taken whilst sampling.

Due to their small size, microfossils can readily be transported and deposited in clasts of original matrix, or as isolated individuals.

When reworked material occurs in the form of isolated individuals, they are usually corroded and less well preserved than contemporaneous fossils, although exceptions do occur. Well preserved Upper Jurassic microplankton have been found in an excellent state of preservation in Eocene (London Clay) assemblages (G.L. Williams, 1964), and reworked Chalk foraminifera are not uncommon at some levels in the Eocene (A.D.K.; Curry, 1952, p. 202). An abundance of reworked well preserved microfossils can dominate over the contemporaneous fossils and by their presence suggest an earlier incorrect age for the assemblage.

If fossils occur enclosed in particles of the original sediment (transported clasts), they may be redeposited in sediments of a very coarse nature; contemporary material being removed by winnowing and sedimentary sorting. Transported clasts are often readily identifiable in thin section.

Microfossil (and also macrofossil) assemblages can therefore be composed entirely of reworked material or a mixture of reworked and contemporary material.

Fuller discussions of this problem are presented by Muir (1967), Tschudy (1966) and Wilson (1964).

Stratigraphic leakage:

This is the direct opposite to reworking, in that it involves the deposition of younger fossil into older beds.

Stratigraphic leakage occurs when fossiliferous sediments are deposited in cracks, fissures, joints and solution channels in older consolidated sediments, e.g. Rhaetic material can be seen infilling crevices in Carboniferous limestone in the Mendips (For details concerning the collecting of fossils from fissures see Kermack, 1965).

Fortunately however, stratigraphic leakage seldom constitutes a serious problem to palaeontologists and is far less common than reworking. It can be guarded against by careful collecting of samples.

Conclusions

When collecting and processing samples for micropalaeontological studies, the various possible sources of error and contamination must be borne in mind and where possible allowed for.

To quote from Funkhouser (1965), "If we find *Ulmus* in the Palaeozoic, we should not rush into print with an early record for angiosperms".

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