MICROPALAEONTOLOGY NOTEBOOK

A note of caution concerning the application of quantitative palynological data from oxidized preparations

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Kerogen extracted from rock samples for palynological studies frequently requires oxidative treatment in order to liberate palynomorphs from amorphous organic matter (AOM). Treatment with nitric acid (70% HNO_3) is sometimes adequate though many Mesozoic organic-rich samples require more severe oxidation to break up clumped AOM. A widely used method involves the repeated oxidation of a preparation in Schulze's solution (70% HNO_3 supersaturated with $KClO_3$), followed each time by rinsing with 2% potassium hydroxide solution (KOH) (Jones, 1994).

The mid-Cretaceous Greenhorn Formation, exposed near Pueblo, Colorado, USA, is composed of interbedded bioturbated limestones and laminated calcareous shales (Cobban & Scott, 1972). In the middle part of the Bridge Creek Limestone Member, kerogen extracted from these two lithologies is markedly different. Diverse, predominantly gonyaulacineaen dinocyst assemblages are found in kerogen from the bioturbated limestone samples. These either require five minutes of nitric acid treatment or no oxidation at all. Most of the AOM is finely disseminated and passes through a standard 10 μm sieve mesh.

Dinocysts in the >10 μm kerogen fraction from the laminated calcareous shale samples are outnumbered by clumped AOM at a ratio of between 10 and 1000:1. Prior to oxidation, gonyaulacineaen and peridiniineaen dinocysts are seen to be present. However, if Schulze's solution and KOH are applied until most of the clumped AOM has disintegrated (a process which takes from 1 to 48 hours), Gonyaulacineae are rare (<5%) in resulting dinocyst assemblages (which are dominated by well-preserved Peridiniineae).

Study of successive oxidation stages (Fig. 1) reveals that a progressive wholesale removal of gonyaulacineaen dinocysts occurs; in contrast, the preferential destruction of thinner-walled taxa (cf. Davies *et al.*, 1982), such as *Oligosphaeridium* Davey & Williams 1966, is not observed. Perhaps the optimum stage of oxidation is reached when there are still several clumped AOM fragments to every dinocyst, such as after the third oxidation of BC50 (Fig. 1). In this preparation, Gonyaulacineae still compose over 20% of the assemblage and dinocysts are sufficiently concentrated to allow the documentation of presence and apparent absence of taxa in that sample. Alternative oxidation techniques employing (1) fuming nitric acid (95% HNO_3) and KOH, and (2) a supersaturated potassium permanganate ($KMnO_4$) solution in an ultrasonic bath, were also found progressively to destroy gonyaulacineaen dinocysts. If kerogen from the bioturbated limestone samples is subjected to repeated oxidations, a similar trend may be observed (e.g. BC48, Fig. 1).

This example of selective palynomorph destruction by oxidation may be particular to the mid-Cretaceous 'black shale' successions investigated by the author. R. Harland (pers. comm.) has conversely found instances of peridiniineaen dinocysts being selectively removed during the

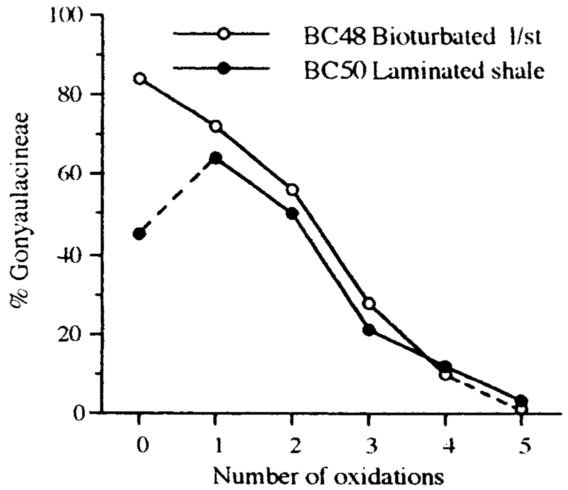

Fig. 1. The proportion of gonyaulacineaen dinocysts in preparations from the same laminated calcareous shale (BC50, Greenhorn bed 106) and bioturbated limestone (BC48, Greenhorn bed 105) kerogen. Oxidations (repeated treatment of the equivalent of 5 g dried sediment in a 15 ml hydrous solution with 60 ml of Schulze's solution and subsequent KOH) are incremented at durations of (BC50): (1) 1 hour, (2) 2 hours, (3) 4 hours, (4) 8 hours, (5) 16 hours; and (BC48): (1) 10 mins., (2) 20 mins., (3) 40 mins., (4) 80 mins., (5) 160 mins. A minimum of 200 dinocysts were counted from each preparation, with the exception of the AOM dominated shale kerogen, BC50 (0), and the impoverished limestone assemblage of BC48 (5); as <50 specimens were inspected from these preparations, the data plots for them may be spurious.

oxidation of Quaternary kerogen. Clearly, it is important for palynologists to assess any detrimental effects of oxidation techniques before attempting quantitative association or palaeoenvironmental analyses of their data. Failure to recognize the control of oxidation on the relative abundance of Gonyaulacineae in dinocyst assemblages from oxidized Bridge Creek shale preparations, could have easily led to erroneous palaeoceanographical modelling of the alternating shale/limestine lithologies.

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