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A new method for bulk palynological processing of oil-contaminated chalks

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INTRODUCTION

The application of palynomorphs in well-site dating and correlation within the Upper Cretaceous—Danian Chalk Group of the North Sea, is currently being investigated at the Geological Survey of Denmark. Due to the relatively low abundance of palynomorphs in the chalks, a large sample size (100-300g) is often needed to obtain representative microfloras. Palynological preparation of large chalk samples does, however, create processing problems, especially if the samples derive from oil bearing intervals. The aim of this note is to describe a method developed at the Geological Survey of Denmark to overcome these processing problems.

PROBLEMS IN THE PREPARATION OF LARGE CHALK SAMPLES.

The problems during the preparation process comprise the following factors:

1. The dissolution of carbonates from 100—300g sized chalk samples with hydrochloric acid causes a vigorous reaction that developes vast amounts of foam. The foam development is usually controlled by spraying alcohol or the more hazardous acetone into the reaction vessel; these solvents reduce the vigorous expansion of the foam by reducing the surface tension of the acid. This procedure, however, demands constant surveillance of the acid treatment, because the effect of the solvents is of short duration. Furthermore, the initial vigorous reaction restricts the use of stronger, more effective hydrofluoric acid concentrations to later parts of the acid treatment process, where the reaction is more calm.
2. In the Danish sector of the North Sea, the main producing reservoir is situated in the Chalk Group. Core samples from the Chalk Group are therefore usually oil bearing and contain variable amounts of heavy hydrocarbons. During the palynological preparation process the presence of heavy hydrocarbons slows down the filtration of the residue from the hydrochloric acid process, because the meshes of the filter cloth become rapidly blocked. In the worst cases the residue on the filter cloth becomes impregnated by the heavy hydrocarbons, and a bituminous mass is formed in the filter funnel. Continuation of the palynological preparation procedure has, under these circumstances, proved futile.

RESOLVING THE PROBLEMS IN THE PREPARATION OF LARGE CHALK SAMPLES.

A method has recently been developed to solve these problems:

100—200ml of household kerosene are poured onto the crushed chalk in the reaction vessel before hydrochloric acid is added; in this way, the vigorous reaction and uncontrollable foam formation is avoided (sufficient kerosene should be added to form a *c.* 1cm thick layer upon the acid phase). Furthermore, it is possible to use concentrated hydrochloric acid from the beginning of the dissolution process, thereby decreasing reaction time.

Shortly after the addition of hydrochloric acid a relatively small amount (in our case *c.* 10ml) of an emulgating agent such as Triton X-100 (obtainable from E. Merck, D-6100, Darmstadt, Germany) is added drop by drop. Triton X-100 is capable of dissolving the heavy hydrocarbon particles into both the kerosene phase and the acid phase.

During the dissolution process, the hydrochloric acid in combination with the Triton X-100 liberates the heavy hydrocarbons from the chalk; these hydrocarbons accumulate at the acid/kerosene interface and are typically captured by the kerosene. This capture leaves clean attack surfaces on the chalk for the hydrochloric acid below the kerosene phase, thereby further speeding up the dissolution process.

After dissolution of the chalk, the vessel contains a light coloured water-soluble acid phase below a dark kerosene phase containing the dissolved heavy hydrocarbons from the sample. This is then poured into a filter cloth, and is stirred with lukewarm water and household detergent (oil soap). It is now possible to filter the combined fluids through a 10 micron filter cloth; a small amount of standard workshop hand rinse (e.g. Swarfega) is added to the filter. The filter is closed with a rubber band, and subsequently rubbed gently until the filter is clean when sprinkled with water. The filter cloth containing the cleansed residue is then submerged in hydrochloric acid in order to dissolve surviving chalk lumps. Henceforth normal palynological preparation procedures can be resumed.

By using the above mentioned modified hydrochloric acid treatment, and by avoiding the use of hydrofluoric acid in the subsequent step (which is possible due to the purity of the chalk), it is possible to produce "ready-to-use" palynological slides from a *c.* 100g hydrocarbon-containing chalk sample after only 2 hours.

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