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The application of fluorescence photometry in the maturity evaluation of dispersed organic matter

A long-wave relative fluorescence intensity method, which was recently developed for the determination of vitrinites in coal, was modified to measure vitrinite particles dispersed in organics rocks. These particles occur in assemblages of dispersed organic matter, consisting of various types of plant and faunal material.
The method was satisfactorily modified and a first attempt to evaluate the maturity of the vitrinite in the succession of sediments in an exploratory borehole was completed.

INTRODUCTION

Organic petrology is a comparatively recent field of science dealing with the microscopically visible solid organic matter assembled in rocks. Optical methods are used to identify organic particles of all types which occur in successions of sediments in boreholes. Microscopic studies of organic matter in source rocks are applied today in oil and gas exploration, the main objectives being:

— characterization and identification of various organic constituents;
— determination of their degree of maturity.

Both objectives are important in analyses of the bulk of organic substances usually present in rocks as the same findings can be obtained from very different combinations of microscopic constituents.

Thoroughly evaluated data may provide useful information on:

— the nature of the fluid generated from kerogen;
— the history of petroleum generation;
— the definition of zones of petroleum generation.

The aim of this paper is to discuss the possibility of using relatively long-wave fluorescence relative intensity (FRI) vitrinite measurements to determine the maturity of vitrinite dispersed in a succession of sediments and to find out whether this method is applicable in the exploration of oil and gas zones.

OBJECTIVES

This is the first attempt to apply long-wave fluorescence intensity measurements to research on dispersed organic mater (DOM) in South Africa. Suitable techniques had to be worked out and the application of the method thoroughly evaluated. The main objectives of the work undertaken were as follows:

— to apply long-wave fluorescence techniques in the determination of DOM maturity;
— to provide a supporting method for gas and oil exploration.

THE ORGANIC CONSTITUENTS OF SEDIMENTARY ROCKS

The major cause of coal evolution and hydrocarbon generation is temperature change. This can be detected by studying changes in DOM from the uppermost sequences of a geological succession to the deeper ones. In this respect the overall characteristics of organic assemblages have to be taken into account.

The composition of DOM is far more complex than that of coal. Even its name is not uniform and different terms such as organoclasts and kerogen have been used by different researchers. The latter term is used mainly by palynologists and has a different meaning than the same term used by chemists. DOM consists of particles which originated from plant material as well as from faunal relics. Amorphous mineral-bituminous groundmass, so-called matrix bitumine, is also included.

Humic and **inertinic** particles, which correspond to woody and coaly material in transmitted light, are exclusively of plant origin and are also called phytoclasts. Vitrinites derived mainly from lignin and cellulose are characterized by their relatively high oxygen content and intermediate reflectance. Inertinites are distinguished by high reflectance and high carbon content.

The **liptinic** group consists of particles that derive from hydrogen-rich plant materials like sporopollenin, cutin, suberine, resin, waxes and plant oils. They are of low reflectance and have fluorescence properties.

Organic **faunal** relics, which are rare in coal, may be abundant in source rocks. They often contain much inorganic material such as carbonates, silica and phosphates in the form of shells and the skeletons of macro- and microorganisms.

The **mineral matrix** is composed of mineral-bituminous groundmass and is amorphous. Vitrinites and inertinites are usually clearly recognizable in normal, white incident light in polished sections of source rocks as well as in assemblages of extracted organic particles, whereas liptinites and faunal relics which are the most important constituents with respect to source rock potential are difficult to distinguish from the mineral groundmass. However, their fluorescent properties make them easily visible under UV and blue irradiation.

Genetically, organic particles in source rocks are heterogenous. They are autochthonous, but a substantial portion of them may be allochthonous reworked material. Some of the organic material may come from the incorporation of cavings during the course of drilling (P. Robert, 1980).

APPLIED METHODOLOGY

So far, vitrinite reflectance has proved to be the most suitable physical parameter for checking the rank of DOM in sediments (P. Robert, 1980). The principles of reflectance photometric techniques used for vitrinite phytoclast particles are the same as, for example, vitrinite measurements in coal. According to P. Robert (1980), there are the following advantages of this method for DOM examination:

— it can be used over a very wide range of coalification, from lignite to graphite;

— it increases with burial depth, which helps non-specialists in the interpretation of results;

— it provides greater accuracy than carbon-ratio data when using DOM for the study of thermal phenomena in geological series.

The disadvantage of the method is difficulty in distinguishing *in situ* vitrinite particles from reworked ones or contaminants for reliable measurements. Also a lack of vitrinic particles occurring in series devoid of a supply of continental DOM (for instance in carbonaceous series) may seriously disrupt the maturity evaluation. Another problem may be caused by the environment of sedimentation, for example, a small amount of darker-than-usual vitrinite in sapropelic facies.

Considering all the arguments for and against, it is commonly held that vitrinite reflectance is an important parameter in DOM determination. For the sake of high accuracy of results, however, it should be supplemented by transmitted light and fluorescence investigations.

FLUORESCENCE MICROSCOPY

Fluorescence microscopy is the newest of the optical methods applied in organic petrology. It was introduced by H. Jacob (1952) who invented the uranyl glass standard used until now, and was utilized by P. van Gijzel (1967) who applied the fluorescence property to coalification studies, K. Ottenjann *et al.* (1974) who measured the max and the red/green ratio *Q* of liptinic substances within a rank spectrum between peat and high volatile Bituminous A, and P. Robert (1979) who classified DOM using fluorescence as the criterion.

At present fluorescence measurements are widely applied inorganic petrology. The techniques used in this method are listed below:

Qualitative fluorescence. This method uses fluorescence for distinguishing fluorescent liptinic/bituminous components from non-fluorescent ones. The lack of fluorescence of lipnite indicates a rank corresponding to > 1.3% Rov. UV irradiation is used for low-rank coals and blue light fluorescence for bituminous coals.

Spectral fluorescence. This method was adopted by K. Ottenjann *et al.* (1974) for analysing fossil materials. The method is based on two main parameters:

— max, i.e., the wavelength corresponding to the maximum intensity;

— the *Q* index, which is the red/green ratio between the relative intensity at 650 nm and the relative intensity at 500 nm.

Quantitative fluorescence. This method is based on the photometric microscopy of liptinite and other strongly fluorescent components. Both UV and blue light irradiation are used for low and high rank coals. As an established standard, the uranyl glass is still used, although intensive investigations into more reliable standards have been carried out. The measurements are taken at a wavelength of 546 nm within the green light range. Observations are carried out in reflected light under oil immersion.

The maturity studies and the recognition of the oil window are based mainly on vitrinite reflectance, although the method is not perfect. The "normal" vertical increase in Rov corresponds to a gradient of about 0.15% per kilometre, at least to a depth of 4000 m (P. Robert, 1988). In such a case, the vitrinite reflectance is still as low as 0.35% at a depth of 1000 m, reaching approximately 0.40% at a depth of 2000 m, 0.50% at a depth of 3000 m and 0.65% at a depth of 4000 m.

Coalification phenomena are closely related to the occurrence and nature of hydrocarbons (M. Teichmüller, 1958). At present the hydrocarbon formation is subdivided into three major stages according to rank (N. B. Vassoevitch *et al.*, 1970):

— below 0.4% Rov — immature and with early methane;

— 0.4 to 1.35% Rov — mature to oil; it is to be stressed that the economic limit between oil and gas reservoirs is placed at approximately 0.70% Rov (M. Teichmüller, 1958; B. Durand, J. L. Oudin, 1979).

— 1.35% Rov — "catagenetic" gas.

Fluorescence analyses are a very useful complementary method in research on organic matter maturation. Modes of hydrocarbon expulsion can, to a certain extent, be noticed under fluorescence examination, which helps to identify the occurrence of exsudatinite, as well as smear films, oil droplets and various other exsudates. The formation of exsudatinite corresponds to the beginning of the oil window. This helps to define the main phase of oil genesis.

Alteration of fluorescence intensity in green light (546 nm) measured on the mineral-bituminous groundmass is also very helpful, especially for distinguishing between the immature, mature and overmature oil stages (M. Teichmüller, K. Ottenjann, 1977). Immature source rocks show a positive alteration (increase of fluorescence intensity during irradiation), mature source rocks show a negative alteration and at the overmature stage fluorescence does not exist any more.

Long-wave fluorescence measurements on vitrinite particles have not yet been used in research on the maturation of dispersed organic matter. This paper discusses an attempt to adapt this technique to the field of oil exploration.

TECHNIQUES DEVELOPED FOR LONG-WAVE FLUORESCENCE INTENSITY (FRI) MEASUREMENTS OF DOM

PRINCIPLES OF FLUORESCENCE MEASUREMENTS

Fossil organic matter up to a certain rank consists of molecules which, when excited by rays of short wavelength, emit rays of greater wavelengths whose spectrum may cover a part of visible light and even extend into the infrared zone (P. Robert, 1988). This property is linked to the occurrence of double bonds $C = C$ (E. Stach, 1969). In fossil organic matter, fluorescence is characteristic of poorly condensed, mainly aliphatic constituents. The fluorescence evolves along with rank and finally disappears at ranks of 1.3 to 1.5% Rov. Vitrinite is distinguished from other organic components by its unique fluorescent character. Its natural wavelength is longer than that of equal-rank liptinites (C. Diessel, 1985). From peat to the brown coal stage, the fluorescence intensity of huminite, which is the precursor of vitrinite, decreases and almost disappears at a rank of ±0.6% Rov. The secondary rise of fluorescence takes place at the stage of low-rank bituminous coal and culminates at a rank of 0.80–1.0% Rov (K. Kruszewska, V. M. du Cann, 1990; M. Sasaki *et al.*, 1990). Thereafter it decreases and finally ceases at a rank of 1.4–1.5% Rov. It must be pointed out that the phenomenon of secondary fluorescence in vitrinite is measurable within the red zone of the light spectrum. This specific feature of vitrinite requires the application of special techniques. The basic equipment consists of a microscope with a mercury short-arc lamps as a reflected light source. The light is filtered through coloured glass which is translucent only to UV or blue light. In the *Zeiss Universal Microscope*, this filter is housed together with the dichroid plate and barrier filter. The emitted fluorescent light passes to the eyepiece which allows observation and choice of the right spot for measurement. Once the measured point has been determined, the fluorescent lights is directed through the interference filter of 600–650 nm wavelength to the photomultiplier which sends impules to the reading box. Results from the reading box are stored and calculated un a computer.

CORRECTIONS APPLIED FOR FRI MEASUREMENTS

To fulfil the requirements for measurements of vitrinite as a component of DOM, the following adjustments to the basic system were made and tested:

— a long-wave system was used, consisting of an excitation filter in the blue light range and having a wavelength of 450–490 nm and an interference filter within the red light range of wavelength 650 nm; measurements were conducted in a dark room;

— a uranyl glass standard was used for measurements;

— the diameter of the measuring field was reduced to 16 μm to allow measurements to be made on the smallest vitrinite particles possible; to cover the widest possible spectrum of specimens, a manually operated measuring stage was installed; one hundred measurements were taken on vitrinite particles in such a way that the whole surface of the sample was scanned.

Table 1

Results of Rov rank and FRI measurements of the succession of samples from the analysed borehole

Depth [m]	Rov rank [%]		FRI [%]
	a	b	
640	0.58	0.51	0.80
720	0.64	0.60	0.80
800	0.63	0.57	0.90
880	0.61	0.45	0.64
960	0.62	0.59	0.67
1040	0.69	0.64	0.71
1080	0.71	0.66	0.44
1160	0.72	0.66	0.45
1240	0.71	0.67	0.40
1280	0.71	0.66	0.40
1400	0.76	0.71	0.26
1520	0.79	0.71	0.22
1590	0.77	0.73	0.26
1675	0.68	0.64	0.25
1774	0.76	0.71	0.31
1825	0.76	0.71	0.30
1875	0.80	0.76	0.25
1973	0.79	0.76	0.25
2076	–	–	–
2173	0.83	0.80	0.19
2272	0.81	0.78	0.21
2375	0.86	0.82	0.19
2905	0.89	0.86	0.18
2920	0.81	0.76	0.33
2975	0.94	0.92	0.21
2995	0.92	0.88	0.42
3047	0.87	0.87	0.23
3087	0.97	0.97	0.15
3104	1.02	1.02	0.23
3125	0.96	0.93	0.24
3160	1.03	1.06	0.14
3180	0.95	0.95	0.32
3220	1.03	1.02	0.29
3418	1.03	1.05	0.31

FRI — Fluorescence Relative Intensity

APPLICATION OF THE METHOD AND DISCUSSION OF RESULTS

The use of the method of fluorescence intensity measurement for DOM maturity determination was tested on a set of borehole core samples provided and prepared by SOEKOR (South Africa). These were collected from the succession of sedimentary rocks at a depth sequence between 640 to 3418 m.

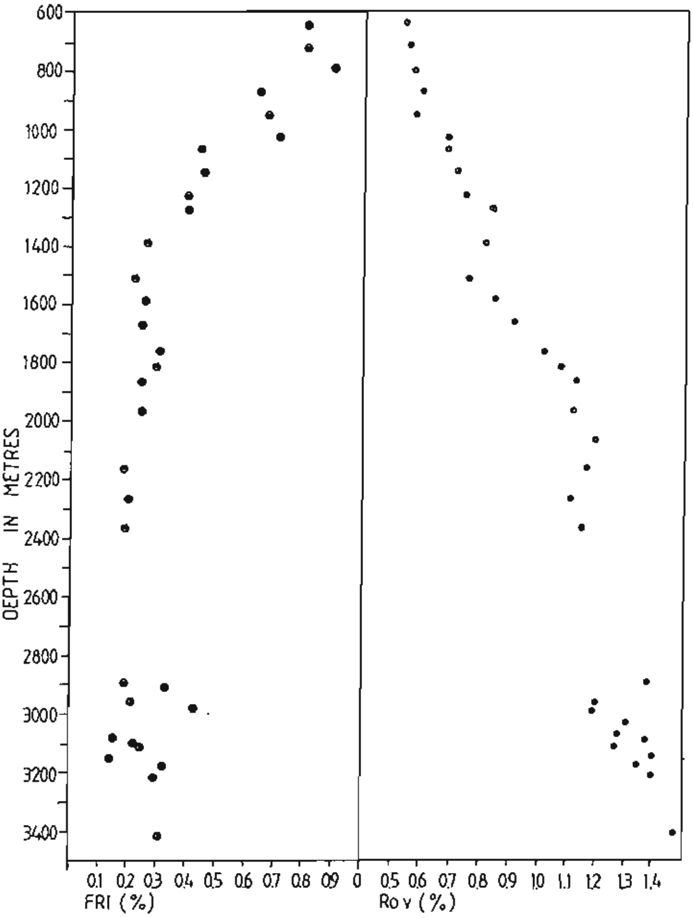

Fig. 1. Changes in the DOM average reflectance and fluorescence values with depth in the rock succession of the borehole core samples provided by SOEKOR
Zmiany średnich wartości refleksyjności i fluorescencji DOM z głębokością

They were prepared in the SOEKOR laboratory using 45% hydrofluoric acid for DOM extraction. Extracted organic matter was separated into two fractions: bituminous-liptinic and vitrinic-inertinic. The latter fraction, which was mounted in synthetic resin, was used for reflectance measurements and subsequently also for fluorescence determination. Altogether, thirty-four samples were examined in both ineident and fluorescent light.

They consisted mainly of vitrinite and inertinite particles with the addition of some spores, bitumens and faunal relics. The particle size of the vitrinite varied from a few micrometres up to approximately 200 μm. Particles of small size prevailed in most samples.

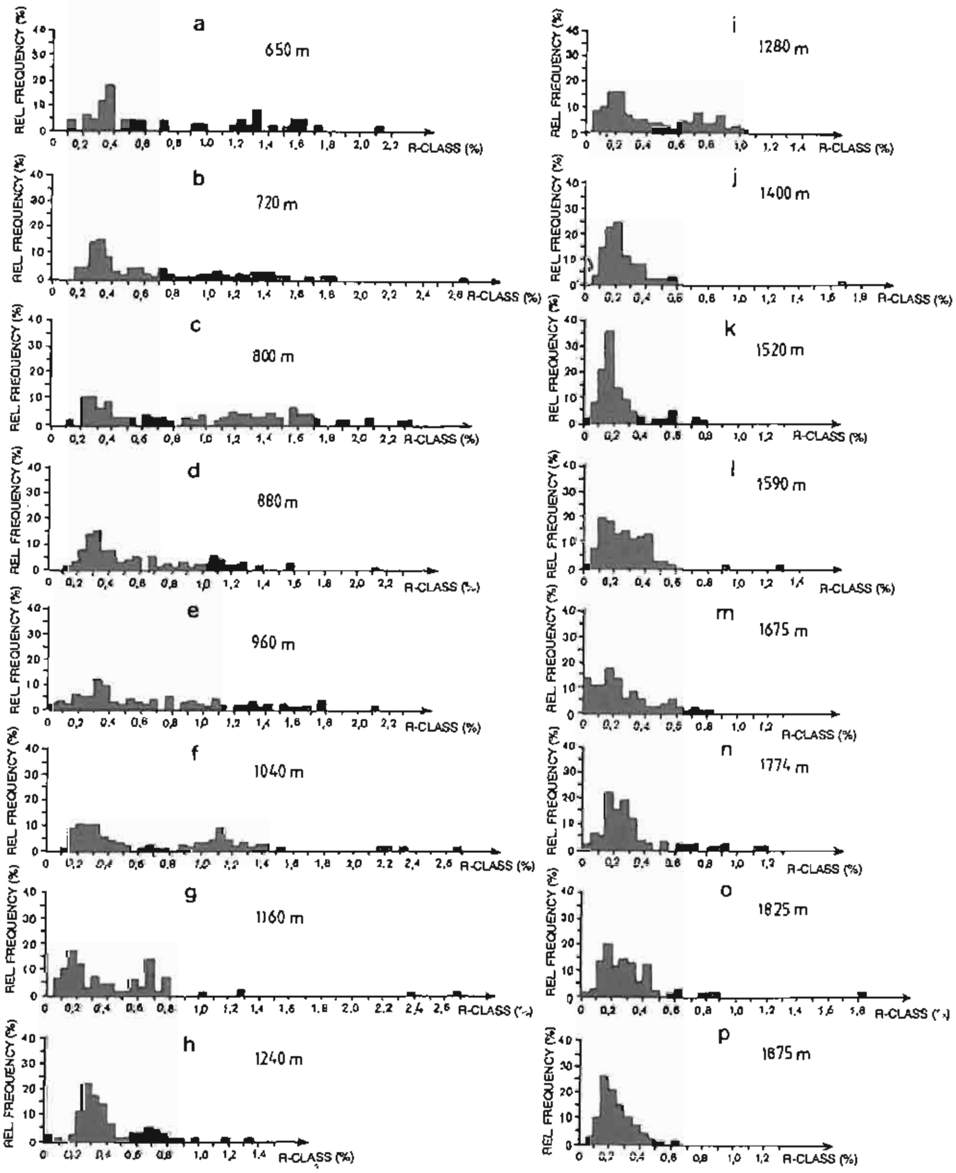

Fig. 2. Histograms of the vitrinite relative fluorescence intensity in analysed samples at the depth of 650–1875 m
Histogramy relatywnej intensywności fluorescencji witrynitu w próbkach z głębokości 650–1875 m

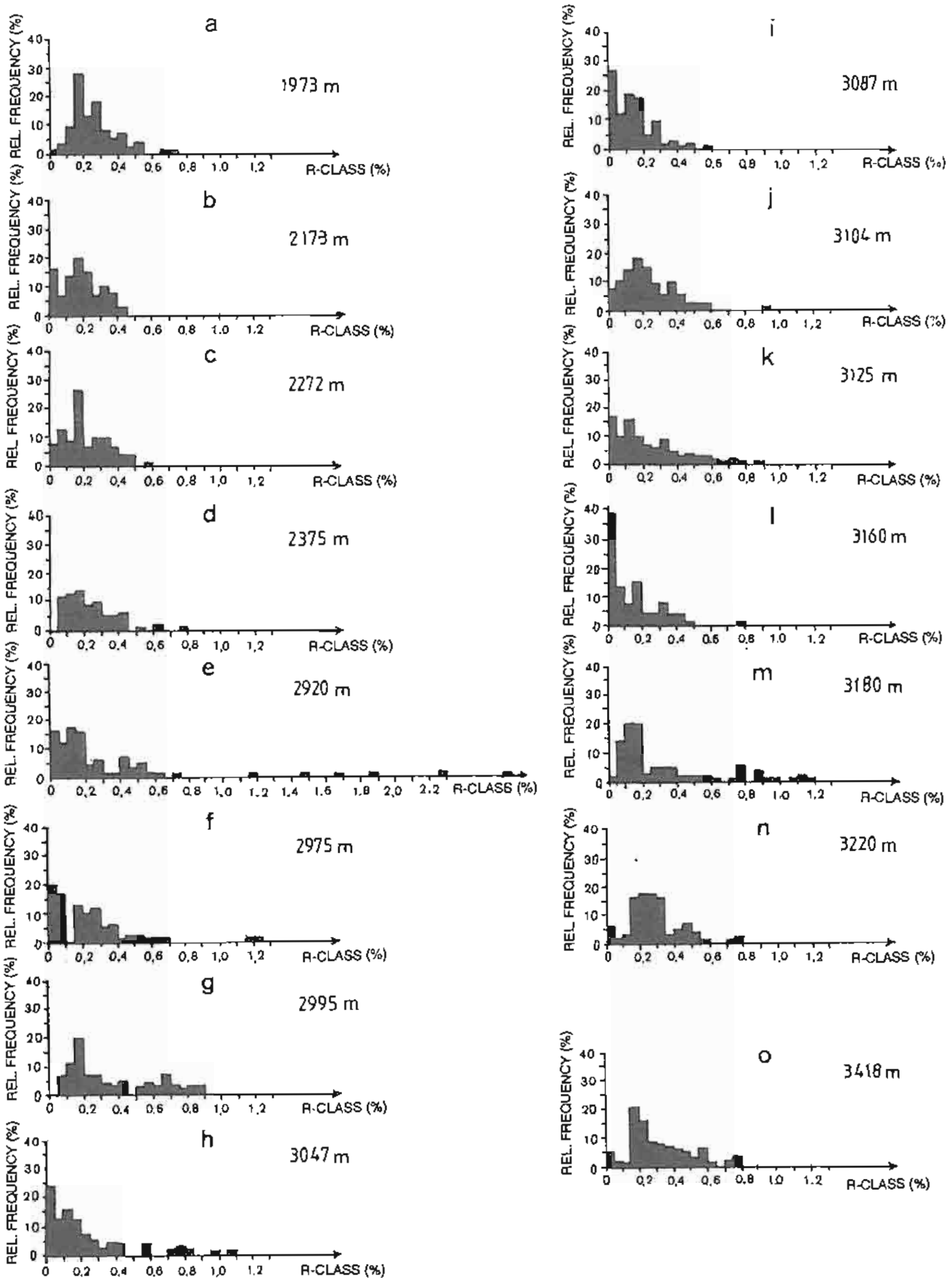

Fig. 3. Histograms of the vitrinite relative fluorescence intensity in analysed samples at the depth of 1973–3418 m
Histogramy relatywnej intensywności fluorescencji witrynitu w próbkach z głębokości 1973–3418 m

VITRINITE REFLECTANCE PROFILES

Vitrinite reflectance was taken as the basis for evaluation of the fluorescence method. The samples were analysed in the Enertek Petrography Laboratory, CSIR and two different ways of obtaining the results were used:

a — only the results obtained from non-oxidized vitrinite particles were considered reliable and used for the calculation of the mean reflectance of the sample; the results obtained from cracked particles, or those with oxidation rims and revealing higher reflectance were discarded;

b — a cut-off point method (K. Kruszewska, 1989) was adapted for the mean Rov calculations; in this case, the mean reflectance plus the cut-off point; only reflectance values lower than that of the cut-off point were used for the calculation of the mean Rov.

The results of all three interpretations are provided in Table 1. The following comments can be made:

1. A comparison between the results achieved with the different methods applied at CSIR revealed that those calculated by the cut-off point method (b) were generally slightly lower to a depth of ±3000 m than those obtained by the selective method (a). The Rov intensities of vitrinites compared from the bottom part of the borehole (more than 3000 m in depth) were of similar values. It must be pointed out that the results from the lowest part of the profile were less regular than those from the upper part. This phenomenon was recorded by both methods. Also, in both cases the borehole reflectance curves had a "normal" sublinear shape.

2. As a result, the whole borehole can be divided into four sequences from the top to the bottom of the borehole:

— the sequence starts with reflectance as low as 0.45% at 640 m; at a depth of 1280–1440 m, the reflectance rises to 0.73–0.75% (Tab. 1, Fig. 1);

— from a depth of 1520 m, the Rov rises more rapidly and reaches a value of 1.04% at a depth 1875 m;

— the sequence between 1875 and 2375 m is characterized by stagnation of the Rov at a similar level and the sequence of samples below, to a depth of 2905 m, was barren.

— the deepest part of the borehole is characterized by scattered vitrinite reflectance.

Generally, she shape of this reflectance curve can be compared to type 4 (intermediate) on the main curve scale (Fig. 1).

FRI PROFILE

The set of samples analysed for vitrinite reflectance was also analysed in fluorescent light. Altogether, 33 samples were investigated (Table 1, Fig. 1).

The following comments on the fluorescence results can be made:

According to mean fluorescence intensities, the whole profile can be divided into three sequences from the top to the bottom of the borehole:

I. The uppermost sequence of the borehole, between 640 and 1400 m, is characterized by the highest mean fluorescence intensity. The mean values are rather scattered, but generally decrease sharply with depth (Fig. 2a–j). The histograms of individual samples usually consist of a main portion of particles which display lower fluorescence intensity.

This part of the spectrum may contain low-fluorescing autochthonic vitrinites such as corpocollinites and some telocollinites, but also pseudovitrinites and to some extent oxidized reworked material. The higher fluorescing part of the spectrum, which consists mostly of desmocollite particles, is much wider and may reach fluorescence as high as 2.3%. Within some of the histograms, additional high-fluorescence peaks appear (Fig. 2a, d, f). The other histograms are composed of a main peak and widely spread high-fluorescence readings (Fig. 2b, c, e). The range of fluorescence intensity narrows with depth and moves towards lower intensities. The deepest histogram of this sequence is already relatively compact and its recorded intensity is limited to a range between 0.1 to 0.6% (Fig. 2j).

II. The next lower sequence is defined between the depths of 1400 and 2375 m. It is characterized by a limited range of fluorescence intensity which does not exceed, or only marginally exceeds, 0.60% (Figs. 2j–p, 3a–d). Consequently, the mean fluorescence in samples is reduced and only in one instance slightly exceeds 0.3% FRI. Very characteristic of this sequence is a slight decrease in values of FRI intensity with depth (Fig. 2a).

III. The sequence below 2400 m down to 2900 m is defined as a hiatus (the samples were barren, with no phytoclasts recorded).

IV. The bottom sequence, between 2900 and 3418 m, is characterized (in a similar way to the reflectance values) by a scattering of mean FRI intensities (Fig. 2d). In two cases, results exceeded mean values of 0.3% FRI, and in one case, even 0.4% FRI (Fig. 2a).

Consequently, histograms of higher FRI mean values were extended towards higher ranges of fluorescence intensities (to the limit of approximately 1.0%). This phenomenon may be related to the occurrenee of secondary fluorescence.

COMPARISON BETWEEN REFLECTANCE AND FLUORESCENCE VALUES

The mean values of both reflectance and fluorescence presented in Figure 1 show some comparable regularities. Generally, reflectance decreases from the top to the bottom of the succession of sediments in the borehole, while fluorescence shows an opposite trend.

The main feature of the fluorescence curve is a significant change in both the shape of the histograms (Figs. 2 and 3) and the rate of FRI decrease with depth at a level of 1400 m (Fig. 2a). The shape of the fluorescence curve is also interesting from a theoretical point of view. According to present knowledge, at the rank level of 0.70% Rov, secondary fluorescence intensity should rise more significantly. This suggests that the long-wave fluorescence in some samples analysed has been destroyed to some extent. There are two possible explanations for this phenomenon, which are not mutually exclusive:

1. The fluorescence might have been destroyed during sample preparation by the use of hydrofluoric acid. To test the extent of the destruction of secondary fluorescence by hydrofluoric acid HF, an experiment was conducted at Enertek on a sample of fresh DNC coal. The coal was cut down to a size of –20 # and treated with a 75% solution of HF for about 60 min. The measurements taken on the sample before and after the treatment showed a very dramatic decrease in fluorescence intensity from 3.13 to 1.66%. This would suggest that different sample preparation techniques should be used in the future.

2. The other possible cause of the low fluorescence performance in the borehole samples analysed is the period of storage. The microscopic slides were prepared in 1981 but the reported information was obtained only recently. After a period the samples must have been

oxidized and consequently their long-wave secondary fluorescence intensities severely affected (K. Kruszewska, V. M. du Cann, 1990).

CONCLUSIONS

Based on the discussion presented, the following conclusions can be drawn:

1. Long-wave fluorescence intensity techniques provided useful information concerning alteration of phytoclasts with depth.

2. To prevent destruction of the secondary fluorescence of vitrinite in DOM assemblages, it is preferable that polished blocks of core rock should be used for microscopic examination. Alternatively, froth flotation processes may be employed. It is of utter importance that samples used for FRI determinations should be as fresh as possible.

3. A cut-off point method may be applied for differentiation between *in situ* vitrinite particles and reworked ones. However more investigations are necessary for a final conclusion.

4. To provide a full-scale evaluation of the FRI method for oil and gas exploration, the continuation of investigations is necessary.

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ZASTOSOWANIE FOTOMETRII FLUORESCENCYJNEJ DO OCENY STOPNIA PRZEOBRAŻENIA ROZPROSZONEJ MATERII ORGANICZNEJ

Streszczenie

W artykule omówiono zastosowanie metody fluorescencyjnej do pomiarów detrytycznego witrynitu wchodzącego w skład rozproszonej materii organicznej w skałach osadowych. Punktem odniesienia dla oceny przydatności metody były pomiary refleksyjności wykonane na tych samych okruchach witrynitu. Celem wyeliminowania z analizy witrynitów utlenionych i wtórnie osadzonych zastosowano dwa warianty metody: wariant konwencjonalny i półautomatyczną metodę skanningu refleksyjności (K. Kruszewska, 1989). Wykazano nieco niższą refleksyjność badaną metodą skanningu w porównaniu z metodą konwencjonalną do głębokości 3000 m, a dopiero wyniki pomiarów próbek najgłębszego odcinka były zgodne, co można by tłumaczyć bardziej jednorodnym charakterem witrynitu potwierdzonym analizą fluorometryczną. W obu przypadkach zmiany refleksyjności miały typowy, sublinearny przebieg z głębokością.

Pomiarów fluorescencji dokonano przy zastosowaniu filtru wzbudzającego niebieskiego 450–490 nm, filtru interferencyjnego o długości fali 650 nm oraz przystawki mikrofotometrycznej z fotopowielaczem, identycznej jak przy pomiarach refleksyjności. W wyniku tych pomiarów podzielono badany profil na cztery odcinki:

I — najwyższy, charakteryzujący się największą średnią intensywnością przy równoczesnym znacznym zróżnicowaniu intensywności w próbce (fig. 2a–f).

II — charakteryzujący się zawężonym zakresem fluorescencji w próbkach; średnia intensywność fluorescencji spada nieznacznie wraz z głębokością;

III — hiatus;

IV — charakteryzujący się nieregularnym przebiegiem średniej fluorescencji z głębokością, podobnie jak w przypadku refleksyjności.

Powyższe rozważania nasuwają następujące wnioski:

— analiza ta może dostarczyć cennych informacji o zmianach zachodzących wraz z uwęgleniem materiału organicznego;

— dla zabezpieczenia badanego materiału przed destrukcją własności fluorescencyjnych należy stosować badania materiału organicznego w szlifach polerowanych lub metodę flotacyjną przy wyodrębnianiu organoklastów ze skały macierzystej;

— metoda skanningowych pomiarów refleksyjności może znaleźć zastosowanie do rozdziału materiału organicznego *in situ* i materiału allochtonicznego w próbce.

Potwierdzenie tych wniosków w praktyce wymaga dalszych intensywnych badań zarówno nad metodą skanningową refleksyjności, jak i metodą fluorometrii witrynitu.