

PALYNOLOGY, PALYNOFACIES AND ORGANIC GEOCHEMISTRY ON THE LOWER CRETACEOUS SUCCESSION (AUDIA FORMATION) FROM EASTERN CARPATHIANS, ROMANIA

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Abstract: This study focused on the Cretaceous black shale from Audia Formation which cropping out in the Eastern Carpathians. This is multidisciplinary approach to the study of some samples from these formation, supported by organic petrography (palynofacies, Thermal Alteration Index – TAI, fluorescence) and organic geochemistry (Total Organic Carbon – TOC). The palynofacies analysis provides evidence that the organic matter is composed of particles belonging to the amorphous organic matter group (in the middle part of the Lower Member) and opaque/translucent phytoclasts which are more numerous in the upper part of Lower Member and the Middle Member of Audia Formation. The palynological assemblage consists of dinoflagellates cysts, some with biostratigraphic importance, and continental palynomorphs such as ferns, Bryophyta and gymnosperms. The TOC data confirm that the samples have a good to very good petroleum potential, and can generate particularly wet gas, condensate or thermogenic gas, and less oil. Type III kerogen is predominant in the samples analyzed, and can be considered thermally mature (in the oil window) based on TAI values and the fluorescence of organic matter.

Keywords: palynology, palynofacies, organic geochemistry, Lower Cretaceous, Audia Formation.

1. INTRODUCTION

In the Eastern Carpathians, the oldest sedimentary deposits (black shales) within the Moldavides units belong to the Audia Formation. These black shales rich in organic matter were deposited during the Lower Cretaceous, being considered rocks with petroleum potential.

The number of studies dedicated to the organic matter (kerogen) of this formation is rather limited. Balteș et al., (1983) noted that the kerogen from the Lower Member with siderites is of the amorphous type, together with various groups of palynomorphs (phytoplankton and spores) and subordinate phytoclasts. At the bottom of the Middle Member with cherts, the kerogen consists of a mixture of Amorphous Organic Matter (AOM), continental phytoclasts and phytoplankton, after which it transforms into a mixture of phytoplankton and continental organic material. This mixture is preserved, also, in the Upper Member of Audia Formation. The authors have estimated the thermal

maturity of the organic matter based on the vitrinite reflectance (R_0). In Lower and Middle Members of Audia Formation, the organic matter is in the mature stage (R_0 between 1.3 – 2.02 %), while the kerogen of the Upper Member is in the immature stage ($R_0 = 0.55\%$), and can generate only biogenic methane. Informations about the organic matter contained in a formation equivalent to that studied in the present paper (Sărata Formation, located in the Vrancea Nappe) was provided by Amadori et al., (2012). The silicified black shales of the Lower and Middle Members revealed a TOC between 0.81 – 1.78% (fair → good petroleum potential), and a kerogen is predominantly type III, and less type II. This organic matter is in the mature stage of the hydrocarbon generation, having a T_{max} between 434 – 442°C. Anastasiu et al., (2013) considers that the organic matter contained in the Middle Member of the Audia Formation is mainly of marine-algal origin (type II kerogen), with a greater thermal maturity (a mean value of the vitrinite reflectance 1.19%). This maturity has allowed the evolution of organic matter

toward a dry-gas window.

The aims of the present paper are, thus, the following: (1) to identify the palynological taxa (continental and marine) and to make biostratigraphical assessments based on these palynological assemblages; (2) to identify the types of kerogen based on optical criteria and to determine its thermal maturity; (3) perform a palynofacies analysis specific for this geological formation; (4) to determine the potential hydrocarbon-generating source rocks (after TOC content).

2. GEOLOGICAL SETTING

The Eastern Carpathians represent a segment (over 600 km long) of the Carpathian tectonic chain. They are mainly composed from sedimentary rocks of Jurassic - Miocene age, deposited in several basins, folded and overthrust on the Miocene sediments of the Carpathian Foredeep.

According to their main deformation phase, the following tectonic units have been recognized in the Eastern Carpathians (Săndulescu, 1984): the Pieninids, the Dacides and the Moldavides (Fig. 1).

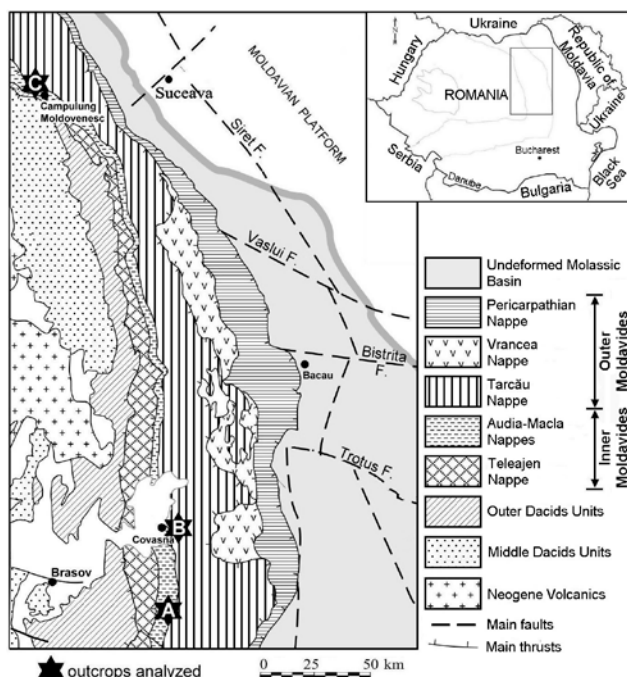


Figure 1. Geological sketch map of the Eastern Carpathians (after Bădescu, 2005), with location of cross-section analyzed.

All geological cross-sections investigated in this paper are located in Moldavides, which represents the largest nappe system of the Eastern Carpathians. Săndulescu (1975, 1984) has divided the Moldavides, based on their tectonic position, into the Inner Moldavides (i.e. Teleajen, Macla and

Audia Nappes), composed of Cretaceous sediments, and the Outer Moldavides (i.e. Tarcău, Vrancea and the Subcarpathian Nappes), which consists of Cretaceous – Cenozoic deposits (Fig. 1).

The Lower Cretaceous organic-rich black shales of the Eastern Carpathians are known from the eastern part of the Inner Moldavides (i.e. Audia Nappe), and from the Outer Moldavides, such as the Tarcău Nappe (Filipescu et al., 1963; Ștefănescu & Micu, 1987; Melinte-Dobrinescu & Roban, 2011), where they belong to the Audia Formation. In Vrancea Nappe, the equivalent of Audia Formation was named by Băncilă (1955), the Sărata Formation, showing some changes in facies within the Upper Member (Grasu et al., 1988).

The Audia Formation has a thickness of about 200 m and has been separated into the following three members (Filipescu, 1955; Melinte-Dobrinescu & Roban, 2011; Roban & Melinte-Dobrinescu, 2013):

- The Lower Member (Upper Valanginian – Upper Barremian) composed of black and grey shales, interbedded with calcareous sandstone and beds or lenses of siderites;
- The Middle Member (Upper Barremian – Upper Aptian), whose lithology is similar to that described above, but which exhibits a significant increase in black siliceous shales and cherts;
- The Upper Member (Upper Aptian – Upper Albian), which contains black shales, as well as sandstone, graywackes, rarely calcareous sandstones and microconglomerates.

3. MATERIALS AND METHODS

Samples location and description of the outcrops. In the present paper, several samples from Audia Formation, collected from the Audia and Tarcău Nappes have been analyzed (Table 1).

In the Câmpulung Moldovenesc area, the Lower Member of the Audia Formation has been identified on the Corlăteni River (noted with C in Fig. 1). Lithologic, this member is composed of black shales with thickness up to 1 m, alternating with calcareous sandstone (20-30 cm thick), with hieroglyphs. Position of the geological strata is approximately vertical.

The Middle Member of the Audia Formation, included in the Audia Nappe, was analyzed from Bota River outcrop (noted A, Fig. 1), a tributary of the Buzău Valley. A detailed description of this member has been provided by Melinte-Dobrinescu & Roban (2011), who describe a sedimentary sequence of approximately 70 m in thickness, consisting of laminated black shales interbedded

Table 1. Location and elemental geochemical analysis of samples from the Audia Formation.

Stratigraphic Units	Outcrops location (Fig. 1)	Sample	TOC (%)	H (%)	S (%)	Petroleum potential after TOC content (Peters & Cassa, 1994)
Audia F., Middle Member (Tarcău Nappe)	B Covasna Valley	P04	0.984	0.686	-	Fair
Audia F., Middle Member (Audia Nappe)	A (Bota River)	P03	1.295	0.564	-	Good
Audia F., Lower Member (Audia Nappe)	C Corlăteni River	P01	1.949	0.912	-	Good
Audia F., Lower Member (Audia Nappe)	C Corlăteni River	P02	3.918	1.085	4.592	Very good

with black cherts. Centimeter-thick strata of breccias and conglomerates with granodiorite clasts are also mentioned. P03 sample (Table 1) derived from the Middle Member of Audia Formation.

From the Covasna Valley, sample P04, assigned to the Middle Member of the Audia Formation from the Tarcău Nappe (noted with B in Fig. 1), was analyzed. Melinte-Dobrinescu & Roban (2011) describe from this member a sedimentary sequence consisting of laminated black shales and levels of lithic sandstones, as well as interbedded black cherts.

Organic matter analysis. The organic matter analyzed from the samples mentioned above was processed using standard palynological techniques (e.g., Batten, 1999). The visualization of palynomorphs was performed with a Leica DM1000 microscope using transmitted white light and blue light fluorescence.

Palynofacies description was carried out on the same microscopic slides used for palynological analysis. The organic particles were classified at the three main groups of kerogen constituents proposed by Tyson (1995), Mendonça Filho et al., (2002), Carvalho et al., (2006), namely: *phytoclads group*, which includes all fragments derived from terrestrial plants (cuticle, tracheids, black woody tissues, charcoal); *palynomorph group*, containing all taxonomic assemblage (spores, pollen, dinoflagellates); and the *Amorphous Organic Matter group*, which refers to all particulate organic components that appear structureless under the light microscope (phytoplankton and bacterially-derived AOM, higher plant resins and amorphous products of the diagenesis of macrophyte tissue).

The determination of kerogen types based on their optical appearance at the microscope was performed according to Combaz (1980), Suárez-

Ruiz et al., (2012), Mendonça Filho et al., (2012).

For the geochemical analyses (C_{org}, H, N, S), a EuroEa3000 EuroVector elemental analyzer was used. The samples were crushed down to a particle diameter < 0.1 mm, after which they were treated with HCl 3N for 24 hours. The remaining material was then washed with distilled water several times so as to remove the acid, and dried in an oven at 50°C.

The thermal maturity of organic matter was determined using Thermal Alteration Index (TAI; Staplin, 1969; Pearson, 1984), and the fluorescence color.

4. RESULTS AND INTERPRETATION

4.1. Palynofacies analysis

Phytoclads, continental and marine palynomorphs, and AOM (gelified and granular) represent the three main groups of the dispersed organic matter analysed from the Audia Formation. The relative frequency of these three groups of organic matter are plotted in figure 2.

The Lower Member of the Audia Formation (identified on Corlăteni River) was analyzed from two samples (P01 and P02), containing different types of organic matter. P02 sample (collected from the middle part of this member) is abundant in granular AOM of marine origin ($\approx 92\%$), being accumulated under suboxic-anoxic conditions, as indicated by its color (dark yellow-brown; Plate I, Fig. 19). The particles of granular AOM display frequent coccoid bodies (bacteria; Plate I, Fig. 19) on their surface, suggesting that it derives from the degradation of phytoplankton due to microbial action. Gelified AOM, phytoclads and palynomorphs have a low frequency within the kerogen of this sample.

Sample P01, collected from the upper part of the Lower Member (Corlăteni River) contains a larger amount of organic matter of continental origin, consisting mainly, of phytoclasts and gelified AOM (Fig. 2). Phytoclasts are most abundant ($\approx 50\%$), being represented by coaly opaque particles and translucent fragments (woody tissue or tracheids). Gelified AOM is almost always associated with granular AOM, in the form of rounded or angular granules (Plate I, Fig. 20).

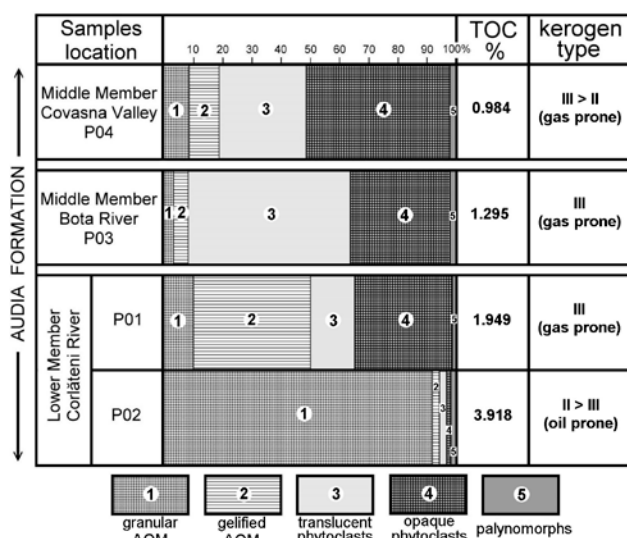


Figure 2. Relative frequency of the three groups of organic matter (kerogen) from the analyzed samples (percentage calculated to Total Sedimentary Organic Matter). The diagram shows TAI values and kerogen type.

The Middle Member of the Audia Formation analyzed from P03 (Bota River) reveals an abundance of continental organic matter, largely consisting of translucent phytoclasts such as woody, in association with numerous yellow-brown fragments with visible structures. To these are added opaque phytoclasts with lath-shaped and granules of brown gelified AOM. The source of marine organic matter is poorly represented, being revealed by rare fragments of granular AOM and dinoflagellates cysts.

The palynofacies identified in P04 sample (Covasna Valley) is roughly similar to that described in the previous sample, coming from the same member of the Audia Formation. The same continental organic matter (opaque and translucent phytoclasts) is prevalent, and to it are added certain groups of granular AOM, which appear associated with small fragments of coaly particles.

4.2. Palynological assemblages and biostratigraphical approach

The palynological assemblage of the Lower Member of the Audia Formation comes from the

Corlăteni River (Câmpulung Moldovenesc). The taxa are predominantly by continental palynomorphs (spores of pteridophyta and bryophyta, pollen of gymnosperms), a similar palynological assemblage being signaled by Balteș et al., (1983) within the same member. Among the dinoflagellates, only one species, *Chlamydomorphella nyei*, identified in sample P01 (Table 2), can be mentioned.

Among the species of continental palynomorphs, the spores of Pteridophyta are predominant, being represented by *Gleicheniidites senonicus*, *Cyathidites australis*, *Clavifera triplex* (cited as reworked in the Șupanu Formation, Comănești Basin; Țabără & Chirilă, 2011), and *Deltoidospora australis*. Less frequent are *Stereisporites antiquasporites* (Bryophyta) and gymnosperms (*Alisporites* sp. and *Cedripites* sp.). The age of the Lower Member which crops out on the Corlăteni River is difficult to specify based on the palynological assemblage, due to the lack of index taxa. However, spore species such as *Clavifera triplex*, *Cyathidites australis*, *Gleicheniidites senonicus* indicate a Hauterivian – Barremian age, as cited in the same stratigraphic interval from the Silesian Unit (Czech Republic) by Svobodová et al., (2011). *Chlamydomorphella nyei* (P01 sample) can indicate an Early Valanginian age (Skupien & Smaržová, 2011), this taxa being mentioned, also, from the younger deposits, respectively Aptian – Lower Albian (Balteș et al., 1983; Svobodová et al., 2011).

Compared with the Lower Member, described above, the Middle Member of the Audia Formation contains a palynological assemblage more abundant in phytoplankton species (Table 2). From sample P04 (Covasna Valley) we could mentioned species such as *Odontochitina operculata*, *Pervosphaeridium pseudhystrichodinium*, *Exochosphaeridium muelleri* a.o., indicating an age not older than Upper Barremian (the first occurrences a species *Odontochitina operculata* are in the lower part of the Upper Barremian; Skupien & Vašíček, 2002; Skupien, 2003; Svobodová et al., 2011). The continental palynomorphs present in this sample are represented mostly by gymnosperms (*Alisporites* sp.).

The Middle Member of the Audia Formation, analyzed from the Bota River (P03, noted with A in Fig. 1) contains a palynological assemblage represented by the cysts of dinoflagellates (*Florentinia* cf. *mantellii*, *Cribroperidinium edwardsii*, *Oligosphaeridium* cf. *complex*, *Scriniodinium pharo* a.o.), fern spores (*Gleicheniidites senonicus*), gymnosperms (*Alisporites* sp., *Pinuspollenites* sp.) and angiosperms (*Stellatopollis dejaxii*).

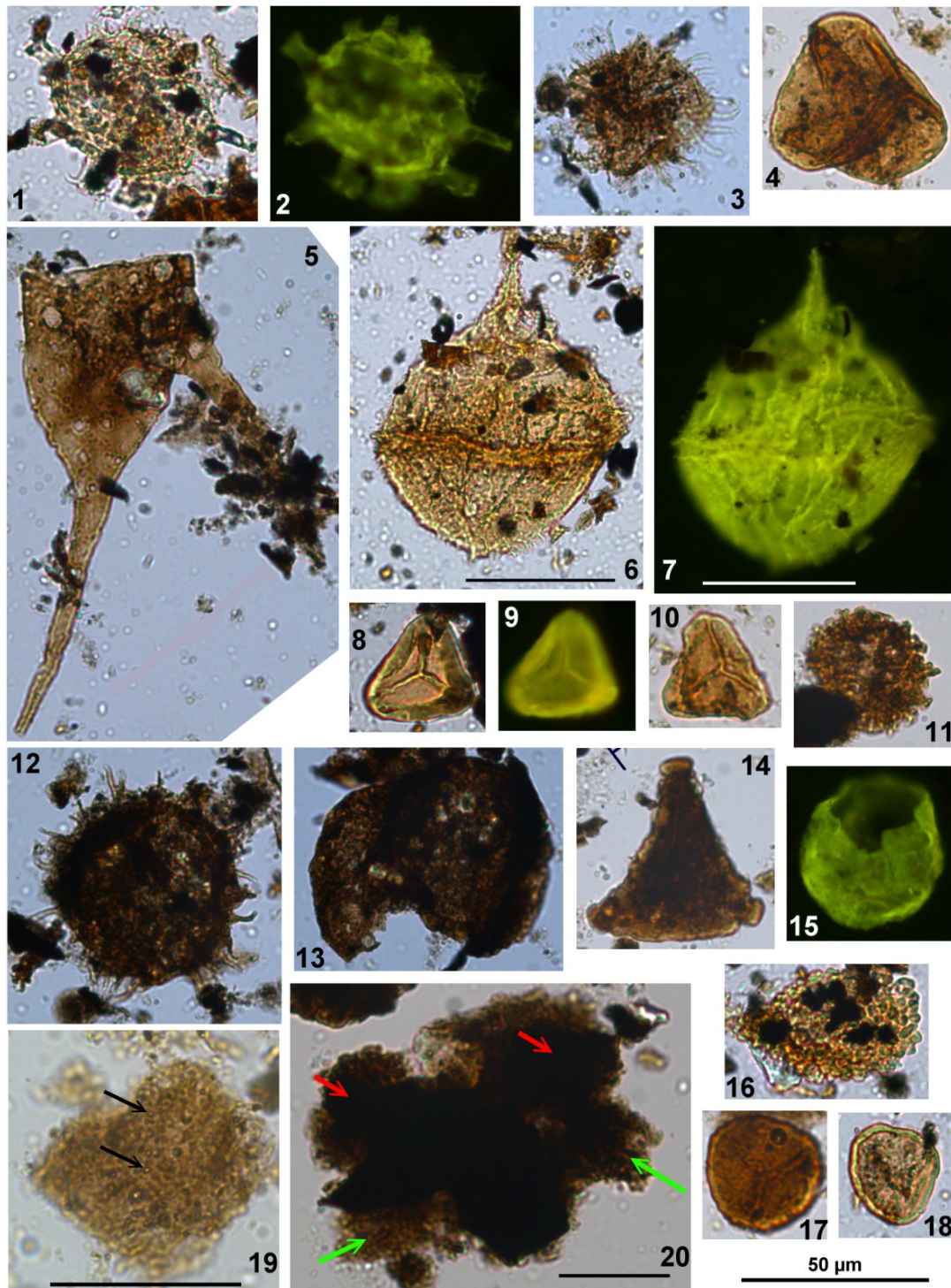


Plate I: 1, 2. (transmitted light and incident blue light, fluorescence) *Florentinia* cf. *mantellii* (Davey & Williams 1966) Davey & Verdier 1973 (P03 sample); 3. *Exochosphaeridium muelleri* Yun 1981 (P04 sample); 4. *Deltoidospora australis* Couper 1953 (TAI = 3-), P02 sample; 5. *Odontochitina operculata* (Wetzel, 1933) Deflandre and Cookson 1955 (P04 sample); 6, 7. (transmitted light and incident blue light, fluorescence) *Cribroperidinium edwardsii* (Cookson & Eisenack) Davey, 1969 (P03 sample) (scale bar: 50 μ m); 8, 9. (transmitted light and incident blue light, fluorescence) *Gleicheniidites senonicus* (IAT = 2+), P03 sample; 10. *Cyathidites australis* Couper 1953 (TAI = 2+), P02 sample; 11. *Chlamydophorella nyei* Cookson & Eisenack, 1958 (P01 sample); 12. *Pervosphaeridium pseudhystrichodinium* (Deflandre 1937) Yun 1981 (P04 sample); 13. *Alisporites* sp. (TAI = 3+), P04 sample; 14. *Clavifera triplex* (Bolkhovitina 1953) Bolkhovitina 1966 (TAI = 3), P01 sample; 15. (incident blue light, fluorescence) *Batiacasphaera* sp. (P03 sample); 16. *Stellatopollis dejaxii* Ibrahim, 2002 (TAI = 2+), P03 sample; 17. *Stereisorites antiquasporites* (Wilson & Webster) (TAI = 3), P02 sample; 18. *Stereisorites* sp. (TAI = 2+), P03 sample; 19. Granular AOM colored dark yellow - brown with coccoid bodies (bacteria, black arrow), P02, Corlăteni river (scale bar: 25 μ m); 20. Granular AOM (green arrow) corresponding to microbial reworking around the gelified AOM (red arrow) (P01, Corlăteni river) (scale bar: 25 μ m).

Table 2. Palynological assemblage identified in Lower and Middle Members of the Audia Formation.

Taxa	Lower Member		Middle Member	
	P01 (%)	P02 (%)	P03 (%)	P04 (%)
Phytoplankton				
<i>Pseudoceratium</i> sp.			4.5	
<i>Coronifera</i> sp.			4.5	
<i>Impagidinium</i> sp.			9.1	
<i>Florentinia</i> cf. <i>mantellii</i> (Davey & Williams 1966) Davey & Verdier 1973			4.5	
<i>Cribroperidinium edwardsii</i> (Cookson & Eisenack) Davey, 1969			13.6	
<i>Cribroperidinium</i> sp.			9.1	
<i>Batiacasphaera</i> sp.			4.5	
<i>Oligosphaeridium</i> cf. <i>complex</i> (White, 1842) Davey & Williams, 1966			4.5	
<i>Oligosphaeridium</i> sp.				9.1
<i>Scriniodinium pharo</i> (Duxbury, 1977) Davey, 1982			4.5	
<i>Odontochitina operculata</i> (Wetzel, 1933) Deflandre and Cookson 1955				9.1
? <i>Cometodinium</i> sp.				9.1
<i>Hystriosphera</i> sp.				9.1
<i>Pervosphaeridium pseudohystriochodinium</i> (Deflandre 1937) Yun 1981				9.1
<i>Exochosphaeridium muelleri</i> Yun 1981				9.1
<i>Chlamydophorella nyei</i> Cookson & Eisenack, 1958	8.3			
Pteridophyta				
<i>Gleicheniidites</i> sp.	33.4		4.5	
<i>Gleicheniidites senonicus</i> Ross, 1949		16.7	9.1	
<i>Cyathidites australis</i> Couper 1953		16.7		9.1
<i>Concavisporites</i> sp.	8.3			
<i>Clavifera triplex</i> (Bolkhovitina 1953) Bolkhovitina 1966	16.7			
<i>Deltoidospora australis</i> Couper 1953		33.3		
Bryophyta				
<i>Stereisporites</i> sp.	8.3		4.5	
<i>Stereisporites antiquasporites</i> (Wilson & Webster) Dettmann, 1963		16.7		
Gymnospermatophyta				
<i>Alisporites</i> sp.	16.7	16.7	9.1	36.4
<i>Pinuspollenites</i> sp.			4.5	
<i>Cedripites</i> sp.	8.3			
Angiospermatophyta				

The age indicated by this palynological assemblage is not older than Lower Aptian, biostratigraphically, most important are the species *Florentinia* cf. *mantellii* (their first occurrences are Lower Aptian; Skupien & Vašíček, 2002; Skupien, 2003). The presence of the species *Stellatopollis dejaxii* suggests a Barremian – Middle Aptian stratigraphic interval, being cited in Lower Barremian in northern Egypt (Deaf, 2009).

The palynological assemblage identified in both samples (P04 and P03) does not include taxa which would allow us to accurately identify the age of the upper part of the Middle Member, species identified being reported, also, from the Upper Cretaceous (Senonian).

Some information about the Lower Cretaceous palaeoenvironment in which the Audia Formation (Middle Member) was deposited can be deducted on account of dinoflagellate species. Thus, we can estimate a littoral environment (*Pseudoceratium* sp.), inner neritic (*Cribroperidinium* div. sp.) and neritic II (*Florentinia*, *Oligosphaeridium*; after Leereveld, 1995; Skupien & Smaržová, 2011). The presence of the genus *Odontochitina* (P04, Covasna Valley) suggests a decrease in the water salinity. The decrease in the amount of granular AOM (marine origin) during the sedimentation of the Middle Member, compared to that of the Lower Member (Fig. 2), indicates a marine regression and the

installation of a marginal dysoxic-anoxic basin (Tyson, 1995).

4.3. Hydrocarbon source potential and thermal maturity

In the evaluation of the hydrocarbon potential of a source rock, the following aspects are taken into account: the quantity of organic matter (expressed through the TOC content), the type of kerogen (oil-prone or gas-prone), and the level of thermal maturation of the organic matter within the sediments (Tissot & Welte, 1984).

The Lower Member of the Audia Formation from the Corlăteni River has a petroleum potential between good and very good (Table 1), the kerogen type inferred after optical criteria being II > III (oil-prone, in P02 sample) and III (gas-prone, in P01 sample). The thermal maturity of the organic matter, inferred based on the TAI, is different in the two samples. In P02, the TAI is between 2+ to 3+ (frequently 3-), indicating a mature stage of hydrocarbon generation (the oil stage generation). The lack of fluorescence of the AOM particles from this sample suggests a maturity of around 0.8 – 1% vitrinite reflectance (R_0), placing it in the late oil window (Smojić et al., 2009; Guler et al., 2013). The thermal maturity of the kerogen from P01 is higher. Thus, based on spore color, we can deduce a TAI between 2+ to 4- (frequently 3), placing this sample within the wet gas or condensate generation stage. The same advanced level of organic matter maturation for the Lower Member of Audia Formation was noted by Balteş et al., (1983), Amadori et al., (2012), Anastasiu et al., (2013).

The organic matter extracted from P03 sample (Middle Member) is abundant in woody tissues with visible structures and coaly opaque particles, characteristic of a kerogen type III (gas-prone), with a good petroleum potential (TOC = 1.295%). The degree of maturation of this kerogen, inferred based on a TAI between 2+ to 3- (frequently 2+ = 0.5 – 0.6% R_0 after Smojić et al., 2009), indicates that the organic matter is in the early oil window, and can generate wet gas or condensate. This maturation stage is also supported by the fluorescence color of the palynomorphs, which is yellow-orange (Plate I, Fig. 9), and equivalent to a R_0 = 0.5%.

The highest thermal maturity of organic matter was observed in the P04 sample (Middle Member, Covasna Valley). The TAI deduced on numerous palynomorphs is frequently 3+ (Plate I, Fig. 13), indicating a stage at the limit between condensate generation and thermogenic gas (dry gas) generation. Kerogen is type III > II (gas-prone),

with a TOC = 0.984%. The phytoclasts, which are abundant in this sample, as well as the palynomorphs (dinoflagellate, spores, pollen), do not display fluorescence, due to the high thermal effect that the organic matter has been exposed to. We believe it is possible for the thermal flow that affected the organic matter from the Audia Formation (Covasna Valley) to have derived from the Miocene volcanism of the Eastern Carpathians, the nearest igneous body being located about 30 km northwest of the outcrop from which the sample was collected.

5. CONCLUSIONS

The present study focuses on the characterization of organic matter (palynology, palynofacies and hydrocarbon source potential) from Lower and Middle Members of the Audia Formation, identified in three outcrops of the Eastern Carpathians. The main conclusions are:

- the palynological assemblage from Lower Member is dominated by continental palynomorphs (Pteridophyta, Bryophyta and gymnosperms), and its age is more difficult to specify because of the lack of index taxa. However, some species of Pteridophyta indicate Hauterivian – Barremian range, as cited within deposits of the same age from the Silesian Unit (Czech Republic). The Middle Member contains a palynological assemblage more abundant in phytoplankton species, *Odontochitina operculata*, *Florentinia* cf. *mantellii* indicating Upper Barremian – Lower Aptian age.

- palynofacies analysis from the Lower Member of the Audia Formation indicate a organic matter of marine origin (granular AOM), accumulated under suboxic-anoxic conditions, as well as a continental organic matter mainly consisting of phytoclasts and gelified AOM, identified at the top of this member. The Middle Member contains mostly continental organic matter (woody tissues, tracheids, coaly opaque phytoclasts).

- the petroleum potential from Lower Member is between good to very good, being able to generate oil, wet gas or condensate. Thermal maturity of the kerogen is high, TAI values and organic matter fluorescence indicating the late oil window. The Middle Member contains a Kerogen type III (gas-prone), abundant in continental organic matter. The TOC content is approximately 1 – 1.3%, with a TAI between 2+ (Bota River) and 3+ (Covasna Valley). The hydrocarbons that can be generated by this type of kerogen are wet gas, condensate and thermogenic gas (dry gas). The Audia Formation which outcropping in Covasna Valley area have a high

level of maturation, due to thermal flow generated by the Miocene volcanism of the Eastern Carpathians.

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