Carpathian Journal of Earth and Environmental Sciences, August 2014, Vol. 9, No. 3, p. 177 - 186

MINERALOGY AND DISTRIBUTION OF THE DETRITAL HEAVY MINERALS IN THE BISTRIȚA AURIE AND DORNA RIVERS ALLUVIAL SEDIMENTS

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Abstract: The Bistrița Aurie and Dorna rivers, tributaries of the longest Romanian mountain river Bistrița, located in the Eastern Carpathians, have been selected for investigations of heavy minerals in alluvial sediments. The investigations comprise basic parameters such as colour, morphology and grain size. However, the major aspect of this study concerns identification of heavy mineral species and their compositions and determining the frequency in the heavy mineral fraction in order to trace their sources and to indicate their possible economic significance. The sampling and method used was in situ panning. Heavy mineral assemblages of the Bistrița Aurie and Dorna rivers are mainly concentrated in grain-size fractions between 500 and 71 μm. The main heavy minerals in the alluvial sediments studied are: garnet (most abundant in the Bistrița Aurie sediments), pyroxene (abundant and present only in the Dorna sediments), manganese- and iron oxides, ilmenite, rutile and pyrite. Other minerals, such as zircon, monazite and titanite, have been identified only as inclusions in various types of minerals. The alluvial sediments of the Bistrița Aurie contain mineral species entirely specific of a metamorphic source, while the Dorna sediments contain minerals typical of both metamorphic and igneous sources.

Keywords: Bistrița Aurie, Dorna River, alluvial sediments, heavy minerals, sediment provenance.

1. INTRODUCTION

The heavy minerals from alluvial sediments have frequently been studied in order to determine the economic potential regarding the presence of gems (Perera et al., 2013), gold and other strategic minerals such as zircon, ilmenite, monazite etc. (Surour et al., 2003, Dill, 2008a; Elsner, 2010). Another approach for heavy minerals research follows the study of their morphology (Moral Cardona et al., 2005; Dill, 2008b), grain size (Joshua et al., 2010) and chemistry, aiming to identify and track the sources of alluvial sediments (Behrends *et al.*, 1999; Macaire, 2013). Provenance analysis of sediments intends to reconstruct their parent-rock assemblages (Joshua et al., 2010). Moreover, heavy metals in river systems could be correlated with the presence of heavy minerals in river sediments (Hochella et al., 2005; Moberly et al., 2009).

The current study aims at investigating heavy minerals from alluvial sediments of the Bistrița Aurie and Dorna rivers in northern Romania. River sediments originate from near surface, exposed igneous, metamorphic and sedimentary rocks belonging to the hydrographic basin. Additional sources of river sediments are mineral components of soils (Joshua et al., 2010).

The purpose of this study is the identification, quantitative analysis and distribution of heavy mineral species, based on their physical properties and qualitative chemical characteristics (EDS spectra) and finaly, determination of likely sources of alluvial sediments of the Bistrița and Dorna rivers.

2. GEOLOGICAL SETTING

The upper Bistrița, upstream of the confluence with the Dorna, called Bistrița Aurie, is part of the

Suceava County, with a length of 70 km. This springs from the Rodna Mountains, while the Dorna River comes from the Călimani Mountains (Donisă, 1963; Donisă & Poghirc, 1968). Through their union, these two rivers form the longest Romanian mountain river, named Bistrița (Rădoane et al., 2009). The main localities crossed by Bistrița Aurie are Cârlibaba, Ciocănești, Iacobeni and Vatra Dornei. The river holds 19 tributaries on the right side and 19 on the left side. The age of the Bistrița and Dorna valleys is considered to be Pliocene-Quaternary (Donisă, 1968).

The Bistrița Aurie crosses the entire Crystalline-Mesozoic Zone of the Eastern Carpathians. This zone belongs to the eastern Getides, within the crystalline basement of the Transylvanian Basin. The Crystalline-Mesozoic Zone consists of several Alpine tectonic units with eastern vergency, deformed during the Austrian tectogenesis (Albian) (Balintoni, 1997; Hârtopanu, 2004). From bottom to top, these are: Infra-Bucovinian, Sub-Bucovinian and Bucovinian nappes (Săndulescu, 1984).

In other words, on the Bistrița Aurie drainage basin surface, medium- and low-grade metamorphic rocks belonging to various metamorphic units crop out. Within the metamorphic sequences of the Crystalline-Mesozoic Zone, the Bretila, Tulgheş, Negrişoara, Rebra and Rodna metamorphic units are individualized (Balintoni, 2005, 2010). Occasionally, Triassic conglomerates, sandstones, limestones, dolomites and lamprophyres veins that penetrate the crystalline rocks appear (Săndulescu, 1984).

The Rebra Unit, a medium-grade metamorphic sequence, represents a typical continental margin metasedimentary pile that contains micaschists with interbedded crystalline limestones, dolomites and amphibolites. The Bretila Metamorphic Unit is a medium- grade sequence dominated by gneisses and amphibolites. The Tulgheş Unit is a low-grade metamorphic sequence and the Negrişoara Unit is represented by a medium-grade succession which consists of a lower paragneiss formation and an upper porphyric metavolcanic unit (Dill et al., 2012).

The Dorna River, with 8 tributaries on the right side and 6 on the left side, drains the northern part of the Dorna Depression, a basin with tectonic and erosive nature, lobate in shape, and being part of the Trans Carpathian Flysch Area (Naum & Butnaru, 1989).

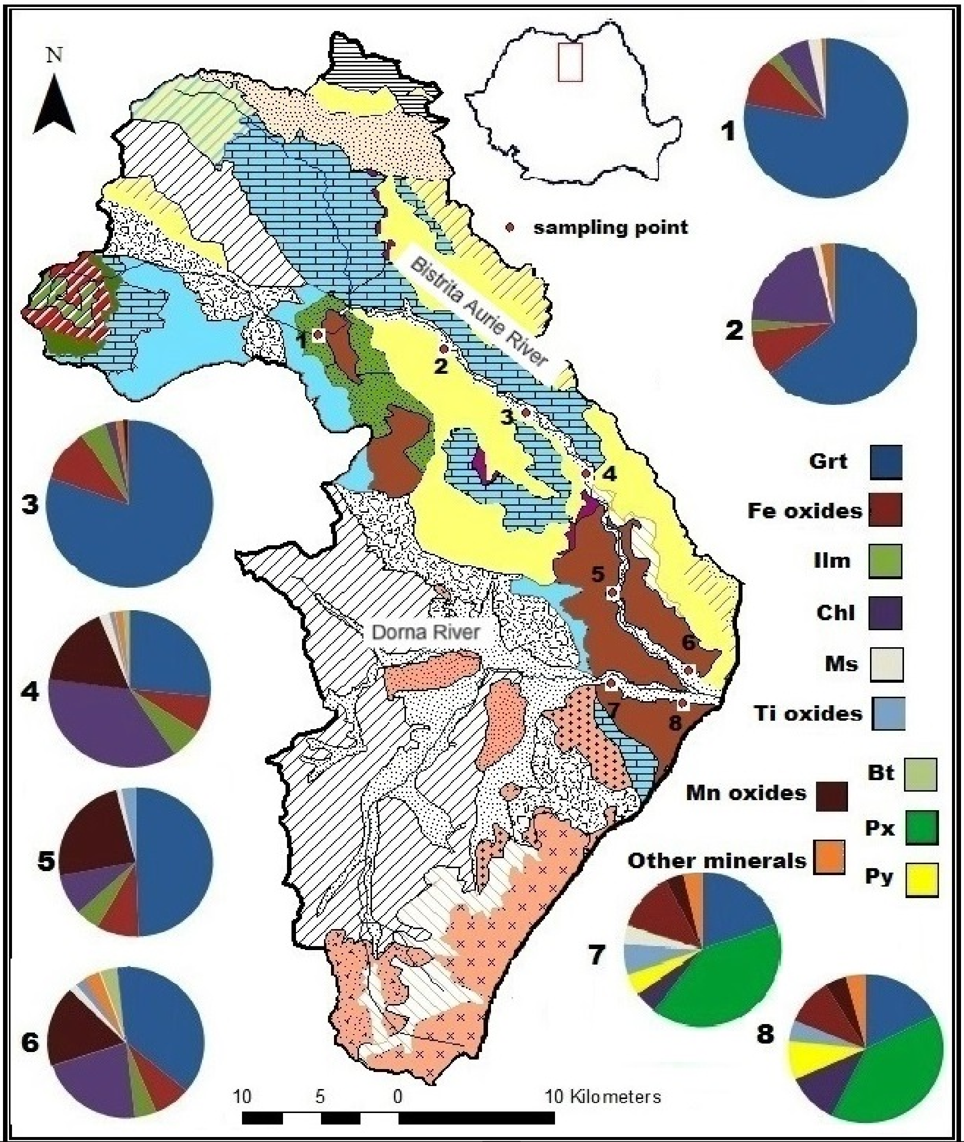
In the drainage basin of the Dorna River, two distinct types of outcrops are found: igneous rocks of the Călimani Complex and metamorphic rocks of the Crystalline-Mesozoic Zone. In addition, isolated sedimentary rocks occur, comprising Cretaceous conglomerates and Eocene limestones. Călimani–Gurghiu–Harghita is the south-eastern, youngest, ~160 km long volcanic range along the Eastern Carpathians (Iancu & Kovacs, 2010). The largest and most prominent volcanic structure is the Călimani Caldera, situated in the north of this area (Seghedi et al., 2005).

The Călimani's petro- and metallogenic field is a young volcanic-plutonic structure, developed between 11.3 and 6.7 Ma, and represented by strato-volcanoes with subcrustal structures; their lithology comprises dacites, andesites, basaltic andesites and pyroclastic flows, diorites and monzodiorites (affected by propylitic and argillic hydrothermal alteration and partially by silicification), sporadically mineralized during Pontian (Stumbea, 2010).

3. METHODS

During the geological prospections, 79 samples have been collected from meanders, from inside bend deposition, gravel bars and alluvial cones. Ten of them have been collected along the Dorna River, 37 along the Bistrița Aurie River upstream of the Ciocănești town and 32 downstream of the Vatra Dornei town. The samples were sieved through a 2 mm mesh; the samples were sifted and submitted to the gravity concentration in a gold pan (as described by Silva, 1986, and Iancu & Mihășan, 1991). Thereby, the initial weight of the sample was reduced from 1.5-2 kg to 80-150 grams of concentrate. The sampling method for the study has the following advantages: it produces a high content of heavy minerals required for the routine analysis, reduces the amount of sample making it easier for transportation, easily eliminates organic material, and limits the costs by reducing or even eliminating the heavy liquids separation.

The heavy mineral concentrates were dried and separated into 8 granulometric fractions. Magnetite and other ferromagnetic minerals were separated from each fraction with a bar magnet. Approximately 500 grains per samples in the 1 to 0.5 mm size fraction were mounted in Epidian epoxy resin, polished and coated with carbon. This granulometric fraction was chosen for gathering more information about inclusions and zonation of minerals and aggregates. For identifying mineral species, a polarizing microscope and the Cambridge Microscan M9 electron microprobe in the Department of Mineralogy and Petrology, Institute of Geological Sciences, University of Wroclaw, were used. The EDS system of the electron microprobe was applied; with beam current 50 nA



Bucovinian, Sub- Bucovinian, Infra-Bucovinian Units
Tulghes Metamorphic unit (Lower Paleozoic)
Tg4-: Quartzite-phyllite Form.
Tg3-Vulcano-sedimentary rhyolitic Form.
Fa- Repedea Series (Silurian):Fântâna Form.
Br- Bretila Metamorphic unit (Precambrian).
Rebra Metamorphic unit (Precambrian)
Rb2- Voslăbeni amphibolite Form.
Rb3-Ineu metaflyschoid Form.
Vcx-Vaser Complex: Rebra and Tulghes Groups not differentiated.
Ro- Rusaia series (Silurian): Rotunda Form.
Mj-Mireaja Form.
PP-Pietrosul Porphyroids. St- Stiol Form.

Lower Volcano-Sedimentary Complex (Pontian)
Andesitic pyroclastic sedimentary breccias.
Pyroxene-, pyroxene-hornblende-andesites.
Intrusive bodies: Hornblende, pyroxene, quartz-andesites.
Lahar flows.
Pyroclastics alternating with epiclastites.
Post Tectonic Sedimentary Cover
Conglomerates and sandstones.
Aluvial deposits.
Bistra sandy flysch.
Flyschoid facies.
Valea Morii shaly-sandy formation and Birtu sandstone with shales (Rupelian).

Figure 1 Geological Map of the Bistrița and Dorna rivers drainage basins (modified after Krautner & Bindea, 2002; scale 1:200000), showing the sample location and the frequency of major heavy minerals from representative alluvial concentrates.

and accelerating voltage 15 kV. BSE images were collected at lower beam currents. Altogether, ten thin sections were analysed with the EDS system.

Additional investigations on the heavy mineral concentrates, in various grain-size fractions (from 0.125 mm to 2 mm), for mineral identification and description of their physical features (e.g., colour, degree of roundness and alteration), were performed with a stereo microscope. For the determination of heavy mineral frequencies, the method of counting grains according to Dryden (1931) was adopted in Luepke (1985), in which more than 200 grains per sample should be identified. Dryden argues that the probable error in the mineral counting shall not be more than 5%. To define accuracy, the probable error (p.e.) of counting n grains in each mineral species was calculated using the following formula:

$$\textbf{p.e.}\ \text{(in no. of grains)} = 0.6745\sqrt{npq},$$

where **n** is the total number of grains identified, **p** is the probability (chance that any grain in the slide will belong to a certain specie) and **q** is the chance that any grain will not belong to such a category (Dryden, 1931).

The diagrams for the main heavy minerals from the representative alluvial concentrates shown in figure 1 were designed in pie.

4. RESULTS AND DISCUSSIONS

The study area was divided into three sub-areas/hydrographic basins, depending on the quantitative participation of major heavy minerals, as follows: (1) the Bistrița Aurie upper basin, upstream of the town Cârlibaba, (2) the Bistrița Aurie lower basin, upstream of Vatra Dornei, and (3) the Dorna basin.

The alluvial sediments from the Bistrița Aurie upper basin (1) are characterized by the abundance of garnets which vary between 79.71 (± 1.33) % and 64.11 (± 1.47) % of the total heavy mineral concentrate. The alluvial deposits of the Bistrița lower basin (2) record a noticeable enrichment in manganese oxides (min. 2.11 (± 1.05) %, max. 23.82 (± 1.55) %). The most abundant mineral from Dorna basin (3) is pyroxene, ranging from 39.72% (±1.46%) to 40.55% (±1.58%).

The heavy minerals encountered in the alluvial sediments of the study river basins are listed in table 1. Heavy mineral assemblages of the Bistrița Aurie and Dorna rivers are concentrated mainly in grain-size fractions between 500 and 71 μm. The grain-size distribution seems to be related to the original size of the minerals in the source rocks. However, taking into account the sampling method, which involved in situ concentration and gravity separation with a golden pan, the grain-size distribution of the heavy minerals is only tentative.

Magnetite was extracted from the alluvial concentrates using a magnet bar. This mineral is usually present in all grain-size fractions.

The magnetite grains from the study sediments present various forms and roundness. For assessing the roundness of grains, the RW (Wadell roundness) visual comparator was used (Cheel, 2005). The magnetite grains range from angular to round in shape. In the samples collected from the Bistrița Aurie upper basin, magnetite is very angular to angular (Fig. 2A). The grains of this mineral become increasingly more rounded as we approach the Vatra Dornei town (downstream) (Fig. 2D). Figure 2 A, B, C, and D show the change from angular to rounded magnetites, when going downstream, that is with increasing distance from the source areas (Mange & Maurer, 1992). This observation confirms that the main source of the magnetite from the alluvial concentrates is located in the upper hydrographic basin. The black metal colour of magnetite crystals from the Bistrița Aurie River is relatively constant in all the samples, while the reddish brown crystals rarely appear. Worth noticing is that, in addition to magnetite, some garnet crystals, mostly translucent, were also separated into the ferromagnetic fraction, probably due to ferromagnetic inclusions.

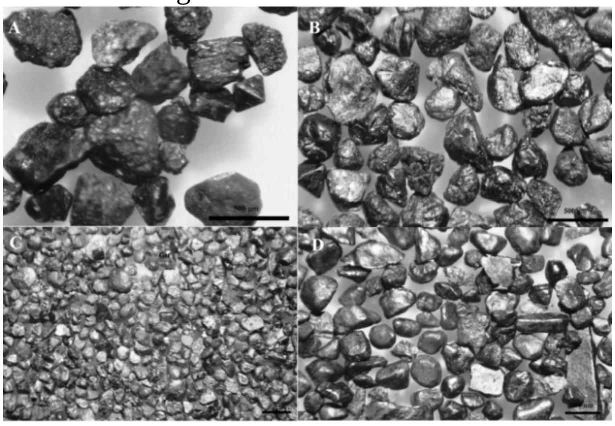


Figure 2. Magnetite grains with increasing roundness from up- to downstream located sample sites; A - sample collected near the springs of the Bistrița Aurie River; B - sample from Ciocănești town; C - sample from Iacobeni town; D - sample from Vatra Dornei town.

Regarding the magnetite from the Dorna River concentrates, its colour ranges from black to reddish and yellow, which suggests another source of this mineral.

As a specific feature, in the ferromagnetic fractions from this area, crystals of dark green pyroxenes (Fig. 6) have been described.

Garnet is the most abundant heavy mineral in the Bistrița Aurie alluvial deposits, especially in the upper basin located upstream of Ciocănești town, where it reaches 79.71 (± 1.33) %.

Table 1. Mineral species in heavy mineral concentrates of representative samples from alluvial sediments of the Bistrița Aurie and Dorna rivers, **x** - single crystal, **o** – inc lusion.

Minerals	Bistrița Aurie upper basin (1)			Bistrița Aurie lower basin (2)				Dorna basin (3)	
	1S	2S	3D	4S	5S	6S	7S	7D	8C
Apatite							**x**		**x**
Augite								**x**	**x**
Biotite	**x**	**x**	**x**	**x**	**x**	**x**	**x**		**x**
Chlorite	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**
Garnet	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**
Enstatite									**x**
Ilmenite	**xo**	**x**	**xo**	**x**	**xo**	**xo**	**xo**	**xo**	**xo**
Hornblende									**x**
Magnetite	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**	**x**
Molybdenite			**x**				**x**		
Monazite			**o**			**o**	**o**		
Muscovite	**x**	**x**	**x**	**x**	**x**	**x**	**x**		**x**
Iron oxide	**x**	**xo**	**xo**	**x**	**xo**	**xo**	**x**	**xo**	**xo**
Manganese oxide			**x**	**x**	**x**	**x**	**x**	**x**	**x**
Titanium oxide	**xo**	**xo**	**xo**	**x**	**xx**	**xo**	**xo**	**x**	**xo**
Pyrite						**x**		**x**	**x**
Pyrrhotite			**x**						
Rhodonite				**x**	**x**		**x**		
Titanite			**o**			**o**			
Zircon	**o**		**o**			**o**	**o**		

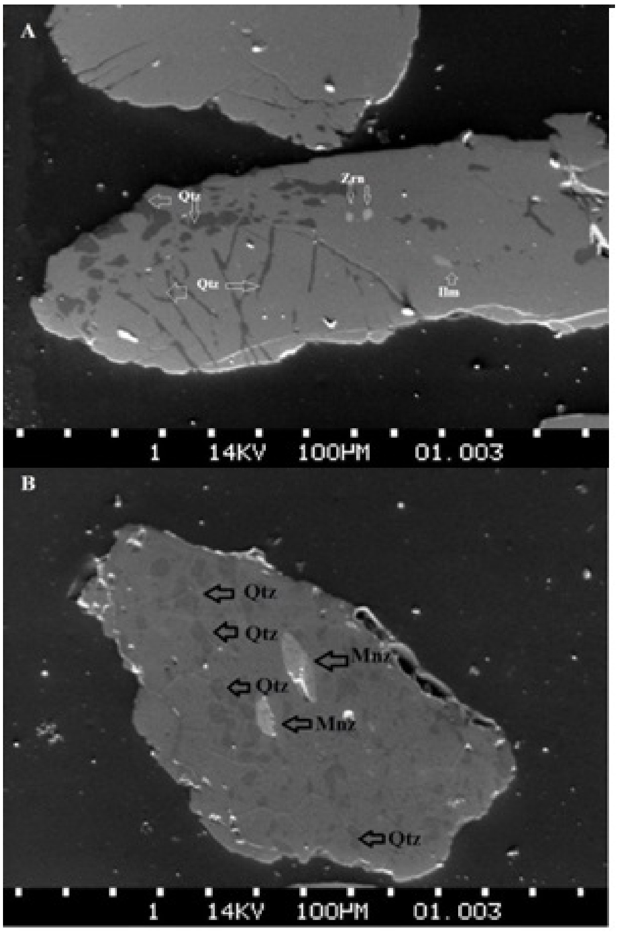


Figure 3 BSE images of two garnets with inclusions. A - inclusions of quartz, zircon and ilmenite; B - inclusions of quartz and monazite.

EDS spectra of garnet from the Bistrița Aurie alluvial deposits highlight four distinct varieties (Fig. 4): almandine-rich (Fig. 4 E), almandine with subordinate grossular (A), grossular-almandine-spessartine (C), and grossular-andradite. These varieties are specific to low- and medium-grade metamorphic rocks. Also, to a lesser extent, a variety with almandine and pyrope end-members has been identified. Based on regional geological literature, the potential source rocks presently exposed for the alluvial deposits contain only almandine, grossular, andradite, and spessartine garnets. The pyrope-rich garnet found in the alluvial sediments of the hydrographic basin of the Bistrița River is a novel element.

Frequently, substitutions of iron with indium, yttrium, silver, samarium, titanium, potassium, etc., in the crystal structure of garnets take place (Keulen et al., 2012). Thus, in the spectra of garnets from the analyzed samples, small peaks of Ti, K, Cr, Zr, Ag and P can frequently be observed (Fig. 4).

Garnet in the Dorna River alluvial sediments appears in all samples, in amount ranging from 17.59 (± 1.14)% to 19.82 (± 1.30)%. Shapes and colours of the garnet from this area are similar to those in the Bistrița Aurie garnets. However, a specific feature of the sediment from the Dorna River is occasionally found bright red garnet (Fig. 7). In the EDS spectra, this type of garnet is richer in calcium compared with garnets from the Bistrița Aurie River, thus possibly derived from high-grade metamorphic rocks (granulites, eclogites) or mafic rocks (e.g. amphibolites), end are specific only for the hydrographic basin of the Dorna River.

Manganese oxides are common; they are missing in some samples collected from the district upstream of Ciocăneşti town, but progressively increase, up to 23.82 (± 1.56)%, while advancing towards this town. Also, they are the main heavy mineral in the alluvial sediment downstream of Ciocăneşti. These oxides were concentrated in the 0.5-1.6 mm range. In the grain fractions below 0.25 mm, the manganese oxides disappear. This group of minerals presents rounded to very rounded shape and they are blackish, brownish and sometime with a bluish tint (Fig. 9A). The source of the manganese oxides in the alluvial sediments of the Bistrița Aurie River is most probably the syngenetic ferromanganese carbonate-silicate deposit, hosted by the black quartzites in the Tulgheș Metamorphic Unit, Ciocăneşti zone (Orata, Orata Mare, Colacu, Oița and Puiu) and Iacobeni zone (Nepomuceni, Arșița and Argestruț).

The oxidation zone comprises an impressive suite of manganese oxides, some of them being described for the first time in this area. The main mineral in this zone of oxidation is represented by psilomelane varieties: ebelmenite, lithiophorite, rankinite, lampadite and asbolane (Sandu, 1960; Hîrtopanu, 2004; Munteanu et al., 2004). As a potential source for manganese oxides of the Dorna alluvial sediments, the Argestruț ore deposit, located in the hydrographic basin of this river, has been identified. The absence or insignificant presence of the silicates such as rhodochrosite and rhodonite in the alluvial sediments of the study area are explained by the fact that the manganese deposits in this area crop out only in the oxidized zone (Fig. 5E).

Iron oxides and hydroxides, except magnetite, occur in the alluvial concentrates in a significant amount of 6.93 (± 0.80) to 10.68 (± 0.96)%. The grains generally range from deep black to gray with a brown metallic lustre. They also form aggregates with quartz and other light minerals.

The EDS spectra, together with microscopic observation, suggest that the main oxide in the river sediments is hematite. For many of the analysed iron oxides, the EDS spectra indicate a well-defined peak of iron with subordinate peaks characteristic of clay minerals. Hematite is also commonly found as inclusions in other heavy minerals from alluvial concentrates. Very often, it forms aggregates with ilmenite and various light minerals.

Among **titanium oxides** identified in the sediments analyzed, ilmenite has the highest proportion, varying widely from 2.44 (± 0.45) % to 19.02 (± 1.22) %.

The concentrate with the highest content of titanium oxides was collected from the Iacobeni town. Among the Ti minerals, apart from ilmenite, rutile/anatase and titanite were identified. Rutile appears as single reddish-brown crystals and in aggregates of ilmenite and quartz.

Frequently, it can be found as inclusions. Anatase was identified only with stereomicroscope and it varies from black to blue-indigo and brown, having perfect bipyramidal forms. Titano-magnetite has been identified only as inclusions.

Iron and manganese oxides from the Dorna alluvial concentrates show similar features to those from alluvial deposits of the Bistrița Aurie River.

Figure 5C, D, E, F show micro-photographs of hematite and manganese oxides and their inclusions. Manganese oxides in the Dorna River sediments have their likely source in the deposits located in the northern part of the Dorna Depression.

Pyroxene is the most abundant heavy mineral in the alluvial sediments of the Dorna River, ranging around 40%, and it is entirely missing from the Bistrița Aurie alluvia. Transparent or translucent crystals have colours from dark green to yellow-green. They are generally euhedral and rarely subhedral, showing that the grains are only poorly mechanically altered, indicating a close source. EDS spectra mostly indicate the presence of augite and, occasionally, clinoenstatite (Fig. 5B). Inclusions in pyroxene comprise a great number of phases, such as ilmenite, titanium-magnetite, hematite, zircon, etc. The most likely sources of the pyroxenes from the Dorna alluvial concentrates are the pyroxene-andesites of the Călimani Volcanic Complex. Another specific feature of the Dorna sediments is the presence of **pyrite** crystals (Fig. 9A) derived from the same volcanic complex. These also occur as well developed cubes or irregular aggregates (Fig. 5A), latten-yellow or tarnished. They were identified in all concentrates from the Dorna Valley, in amount ranging from 4.48 (± 0.66) % to 8.31 (± 0.82) %. Pyrite was noticed in the Bistrița Aurie sediments, but in trace amounts, and only in two samples.

Other minerals occurring in smaller proportions in the alluvial sediments of Bistrița Aurie are chlorite, muscovite, biotite, and apatite. Most of the EDS spectra for chlorite indicate the presence of a retrograde variety after biotite. Also, upstream Ciocănești and at this town, as well as near Dorna town, molybdenite has been found.

In the sediments of the Dorna River, in smaller proportions, the following minerals have been described: hypersthene, hornblende, ilmenite and biotite.

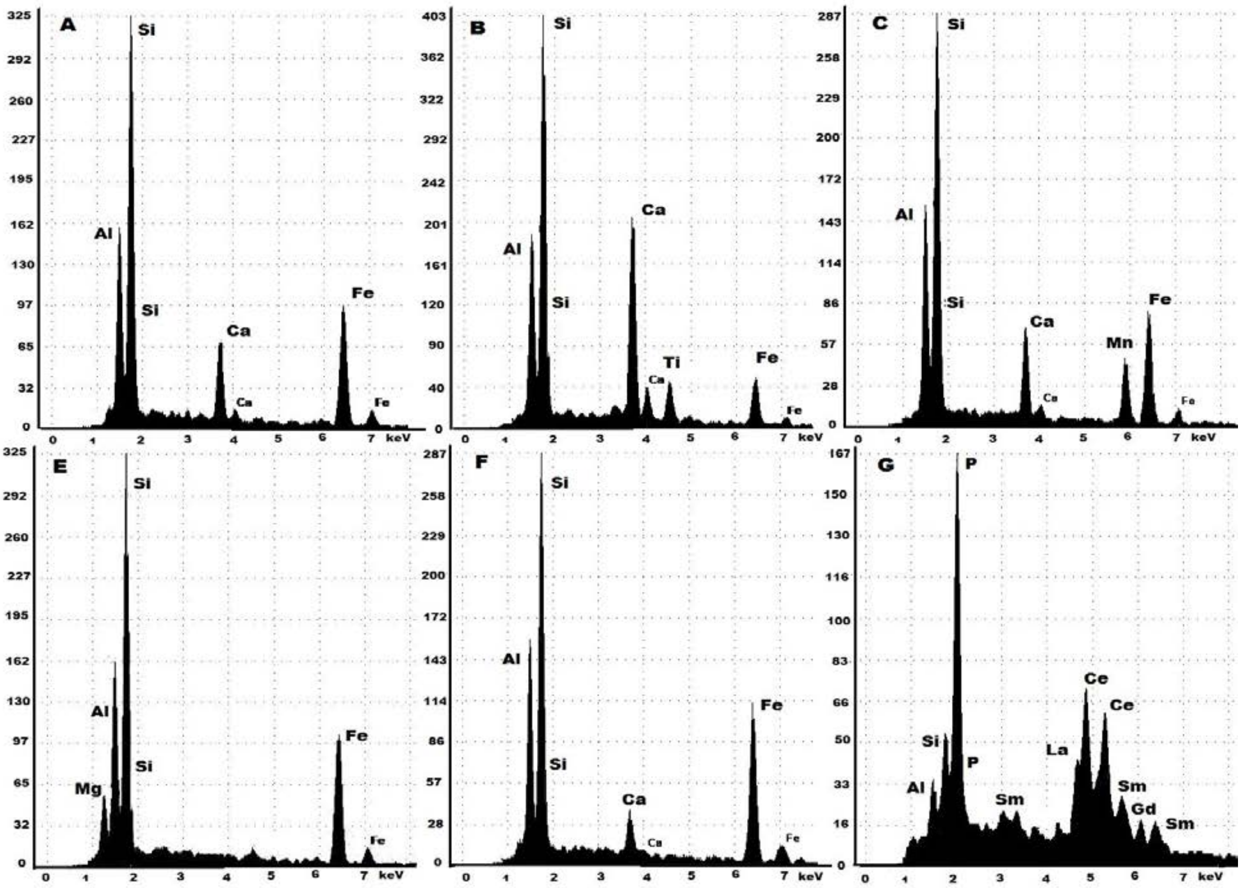


Figure 4 EDS spectra of various garnets from alluvial sediments of the Bistrița Aurie River, A - almandine with subordinate grossular, B - grossular-andradite, C - grossular-almandine-spessartine, D - almandine and pyrope, E almandine-rich, F - inclusion of monazite in garnet.

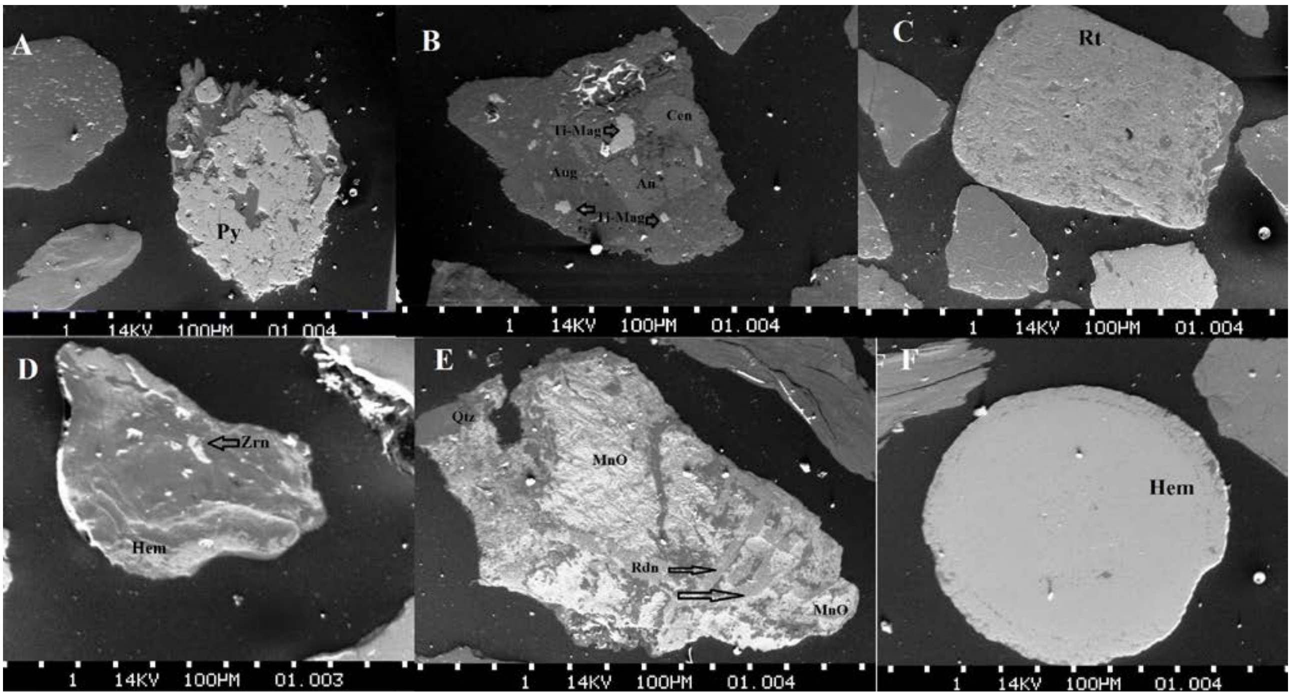


Figure 5 BSE images of some mineral grains from the river sediments of the Dorna River; A - pyrite, B - augite, clinoenstatite, ilmenite and anorthite aggregate, C - rutile, D - hematite with inclusions of zircon, E - rhodonite and manganese oxide aggregates, F - concentric form of iron oxide.

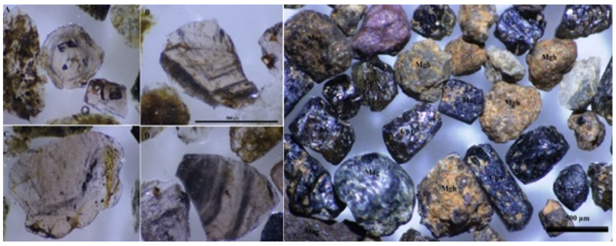


Figure 6 Zoned garnet crystals in thin sections (binocular microscope image, on the left), and ferromagnetic fraction of a concentrate from the Dorna River sediments (on the right side); (500 µm fractions).

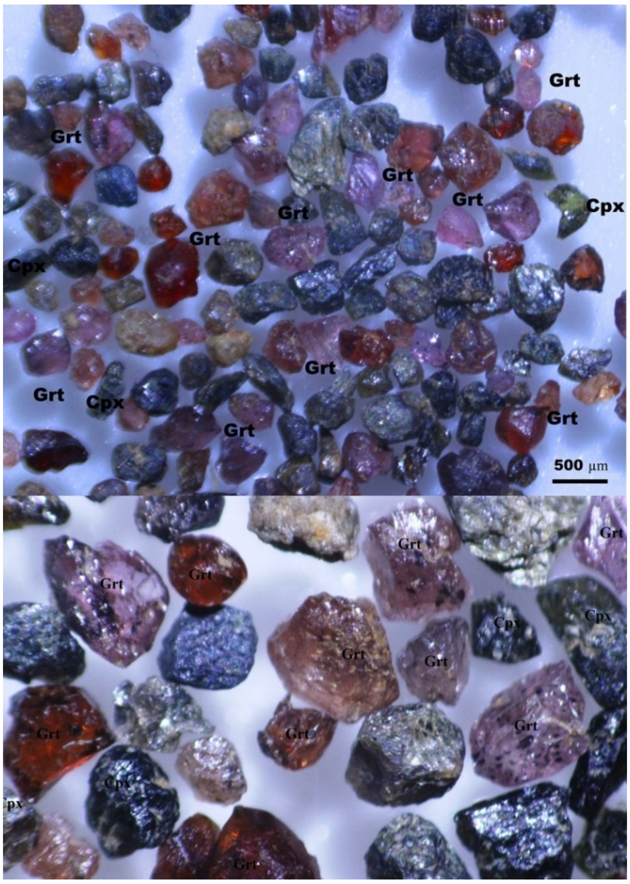


Figure 7 Garnet crystals from alluvial deposits of Dorna River (500 µm fraction)

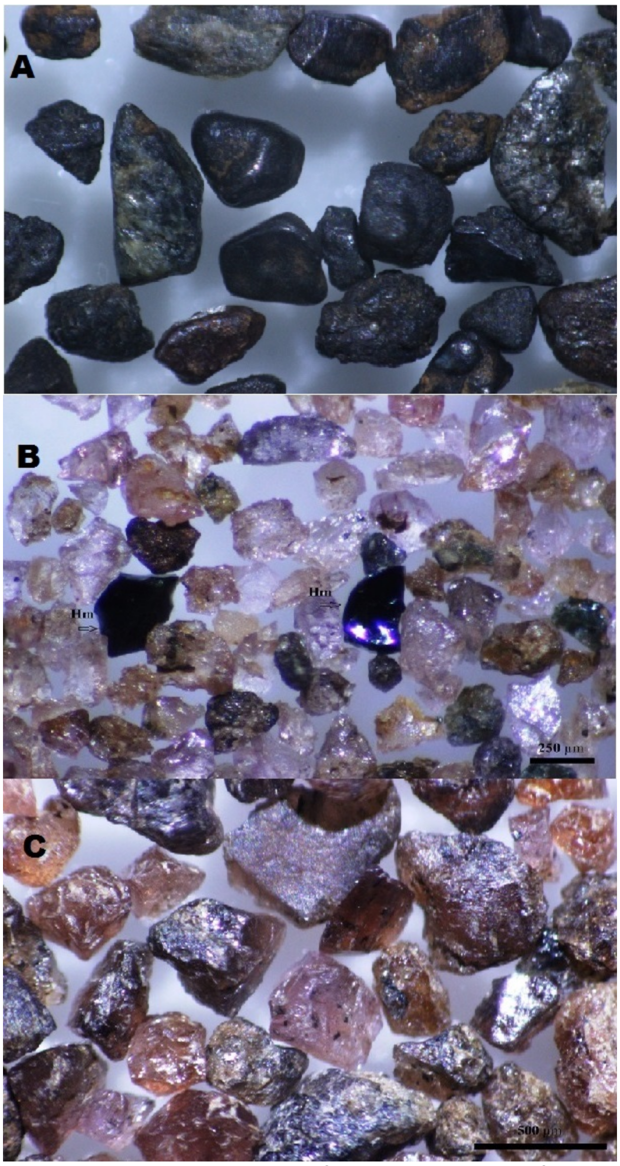


Figure 8 A - Crystals of manganese oxides from the Bistrița Aurie alluvial sediments (500 µm fraction); B, C - (binocular microscope image) Garnet crystals from the alluvial deposits of the Bistrița Aurie River (250 µm and 500 µm fractions, respectively).

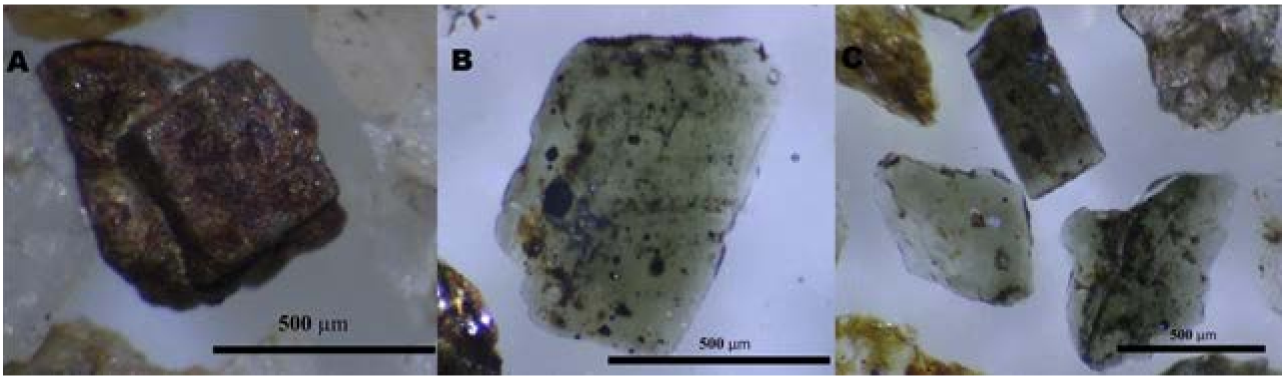


Figure 9 A - Pyrite crystal from the Dorna alluvial sediments; B, C - Pyroxene crystals in thin sections (binocular microscope image).

Zircon, monazite and titanite have been identified in abundance, but only as inclusion form, which indicates the likely presence of such minerals in the grain-size fractions smaller than 0.5 mm.

Among the light minerals ascertained in the alluvial concentrates, quartz, anorthite, albite and K-feldspar can be mentioned. Most of the light minerals have inclusions of heavy minerals, and this is why they are present in the concentrates, varying from 5.47 (± 0.29) to 12.53 (± 0.96) %.

5. CONCLUSIONS

The study of heavy mineral contents in the alluvial sands of the Bistrița Aurie and Dorna rivers revealed that heavy mineral assemblages of both the rivers have been found to be concentrated mainly in grain-size fractions between 500 and 71 μm. The most abundant heavy mineral in the Bistrița Aurie alluvial deposits is the pink garnet, identified for the most part as almandine-rich type; it ranges from 64.11 (± 1.47)% to 79.71 (± 1.33)% of the total heavy minerals concentrate. On the other hand, the garnets in the Dorna sediments occur in lower proportion, between 17.59 (± 1.14) % and 19.82 (± 1.30) %. Their features in the deposits of both rivers are similar, except the presence in the Dorna sediments of a red garnet richer in calcium. The manganese oxides, described in the lower Bistrița Aurie and Dorna, have also different sources for each river basin. They are concentrated only in grain fractions higher than 0.25 mm, most likely due to higher susceptibility for weathering of the aggregates. The source of the manganese oxides in the alluvial sediments is interpreted to be the syngenetic deposits hosted by the black quartzites of the Tulgheș Metamorphic Unit.

However, the alluvial sediments from the Bistrița Aurie and Dorna have different ore deposits as sources for manganese oxides. A particular feature of the Dorna alluvial heavy minerals is the ferromagnetic fraction, which is visibly affected by alteration. Also, besides the magnetite in the ferromagnetic fraction of the Bistrița heavy minerals, translucent (probably due to the inclusions) garnet crystals were separated. In contrast, in the Dorna sediments, a variety of dark pyroxene was described in the ferromagnetic fraction. Summing up, the two studied river basins have, besides obvious similarities, also specific differences in their heavy mineral assemblages indicating different sources of the detrital material.

ACKNOWLEDGMENTS

The authors are greatly thankful to Paweł Matz for making the thin sections in the Polishing Lab of the Institute of Geological Sciences, University of Wroclaw, and to Jacek Ćwiąkalski for his assistance at the electron microprobe analysis. The project was supported from the CEEPUS Network, CIII-RO-0038-09-1314-M-71093.

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Received at: 19. 02. 2014
Revised at: 21. 05. 2014
Accepted for publication at: 22. 05. 2014
Published online at: 28. 05. 2014