Carbonate filler resources of the Bjelopavlići area, Montenegro

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Abstract

Carbonate filler is an important mineral commodity, with rather diverse industrial applications. The Bjelopavlići area in Montenegro is rich in chemically pure limestone, which was preliminary explored in the field, and further its mineralogical, petrographic, geochemical and technological properties were studied. The obtained results have proven the high quality of limestone from the studied deposits, as it can be used for paints and varnishes, rubber and plastics, metallurgy, foundry, sugar, fertilizers and animal feed industry. Several deposits have very pure limestone, that can be also used for paper, glass, pharmaceutical industry and soil neutralisation. The potential resources of high quality limestone are around 23 million tons.

Keywords: limestone, filler, geochemistry, application, resources.

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The carbonate resources of the Bjelopavlići area (Fig. 1) comprise dimension (decorative) stone, aggregate rock, and filler grade carbonate. Their unique feature is that all those industrial types of stone are made of extremely pure limestone, which means that all fully and/or partially explored and analysed deposits can be used as source of filler, even for very demanding markets.

The most important deposits of dimension (decorative) stone in Montenegro are located in the region of Bjelopavlići (area of Danilovgrad and Spuž), where eight deposits have been explored: Visočica, Maljat, Klikovača, Vinići, Radujev krš, Suk, Slatina and Jovanovići, with proven ballanced geological reserves of over 23 Mt. The centre for stone processing is in the nearby town of Danilovgrad. One aggregate rock quarry named Sađavac was also explored. All these deposits were preliminary explored in the last five years as potential source of calcium-carbonate filler [1–4], including several new occurrences: Lalevići, Pješivački do and Mali Garač. Possibility of using limestone from the Visočica deposit in paper production was already studied [5]. The goal of this paper was to characterise and prove the high quality of studied limestone as potential source of filler.

Geological background

The most significant deposits of high purity limestone have been discovered within the Upper Cretaceous carbonate sediments [6] of the High Karst zone (the Bjelopavlići area). High carst covers the central and southwestern parts of Montenegro. This tectonic unit consists of two sub-units: Old Montenegrin and Kuč thrust. Both units, which are separated by a regional dislocation known as Kuč thrust, have a very complex tectonic structure.

Limestones of Senonian age, present in the coastal and central part of Montenegro, were formed mostly on carbonate platform, and consist of bedded to massive limestone, dolomitic limestone and dolomite, with rudists and foraminiferae. Bedded to massive limestone is predominantly bioclastic floatstone to bioclastic grainstone, occassionally with small intraclasts (Visočica), wackestone-packstone (Klikovača) and very subordinately mudstone as cement (Visočica).

High quality Senonian limestone is underlain by Turonian limestone and dolomite, and overlain by Paleogene flysch of the Zeta valley, comprised of marlstone, shale and sandstone with lenses and layers of breccia and conglomerate [6].

EXPERIMENTAL

Samples for mineralogical, geochemical and technological analyses were collected from each deposit, taking into account the differences in macroscopic composition of stone in order to be fully representative. Samples from each deposit weighted 80–100 kg. Representative rock pieces were taken to prepare thin sections for optical microscopy and scanning electron microscopy, and the remaining material of each sample was crushed in laboratory jaw crusher Denver Model Colo 4"x6". After splitting, 5 kg of material was taken

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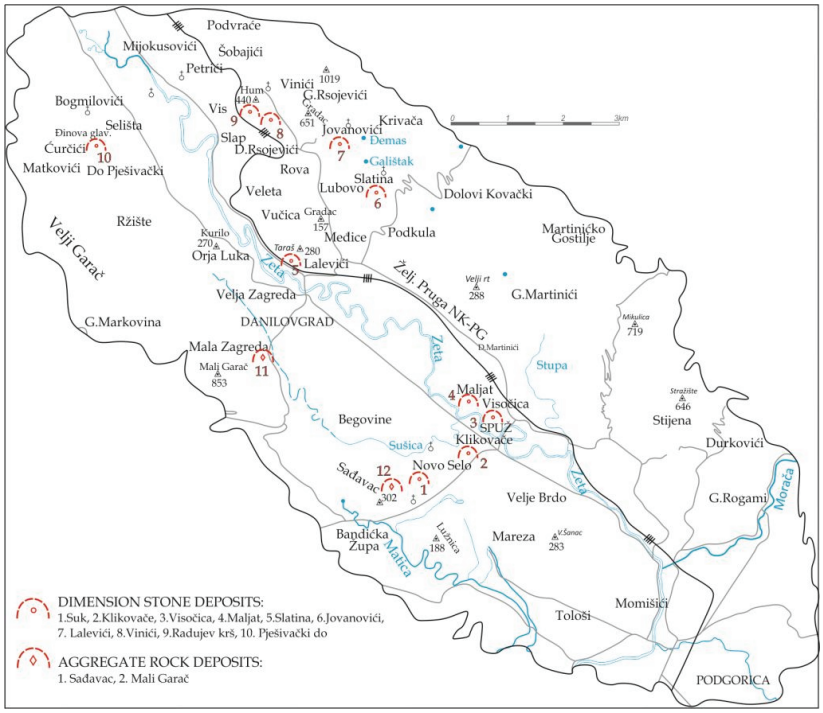


Figure 1. Orientation map with studied limestone deposits.

for laboratory analyses, pulverised in agate stone pulveriser "KHD Humboldt Wedag" and packed in plastic bags and properly marked. Duplicate of each sample has been kept at the Geological Survey in Podgorica.

Mineralogical and technological studies, either on rock pieces or powder, included optical microscopy, X-ray powder diffraction (XRD) and scanning electron microscopy (SEM), differential thermal and thermogravimetric analyses (DTA and TGA), determination of moisture, whiteness, specific weight, oil absorption, water absorption, and pH determination. For XRD and SEM analyses two samples were taken from Visočica deposit and Klikovača deposit.

XRD analyses were performed on Ital Structure APD2000 diffractometer with 2θ range of 5–60°, step 0.02°, and retention time of 0.5 s, using CuKα 1.54178 Å). SEM analysis were done using a JEOL JSM-6610LV scanning electron microscope (SEM) coupled with energy-dispersive X-Max Large area analytical silicon drifted spectrometer (Oxford). The samples were covered by gold using a sputter machine type BALTEC-SCD-005. The analyses were done under acceleration voltage of 20 kV, a beam current of 20 nA and a spot size of 1 µm. Tungsten filament was used as a beam source. Appropriate internal and external standards were used for the analyses. Detection limit for most elements was ~0.1 %.

DTA and TGA analyses were done on all samples using Netzsch-simultaneous thermal analysis – STA 409 EP, heating speed of ΔT = 10 °C/min, in temperature interval from 20 do 1000 °C. Sample mass for analyses was 100 mg.

Geochemical analyses were done at AcmeLabs in Vancouver, Canada. 250 g of rock samples were previously crushed, split and pulverised to 200 mesh. Macro-elements were analysed by XRF (including Lecco C and S) employing fusion techniques to completely decompose the material and provide total element concentrations. Trace elements were analysed by ICP-MS method after multi-acid digestion. Detection limits are given in Table 1.

Table 1. Detection limits of analytical methods

Element	Detection limit, %	Upper limit, %	Element	Detection limit, mg/kg
SiO_2	0.01	100	As	0.5
TiO_2	0.01	50	Be	1
Al_2O_3	0.01	100	Cd	0.1
Fe_2O_3	0.01	100	Co	0.2
CaO	0.01	100	Cu	0.1
MgO	0.01	100	Hg	0.01
Na_2O	0.01	100	Mo	0.1
K_2O	0.01	100	Ni	0.1
MnO	0.01	93	Pb	0.1
P_2O_5	0.01	100	Se	0.5
Cr_2O_3	0.01	10	Sb	0.1
LOI	0.10	–	Th	0.2
C_{total}	0.02	100	U	0.1
S_{total}	0.02	100		

Determination of whiteness was done on 20 g of each sample previously pulverised to 100 % below 63 μm and compared to standard whiteness MgO = 100 %. Oil absorption was analysed on dried 5 g samples with flaxseed oil with specific mass of 0.928 g/cm^3, while water absorption was done on dried 20 g samples with destilled water. pH values were determined on 10 g samples of limestone mixed in 100 cm^3 vessel with destilled water for one hour. After that pH was measuerd by CONSORT C830 instrument, which was previously calibrated to pH 7 and 10 using standard buffer solutions for corresponding pH values. Moisture was determined after heating to 105 °C till constant mass, specific weight was determined using pycnometer in xylene.

RESULTS AND DISCUSSION

Mineralogical and petrographic analyses revealed that studied limestone differs in structure and texture, with variations from boundstone, rudstone and floatstone, to grainstone and, subordinately wackestone, packstone and mudstone (Fig. 2).

SEM analyses revealed that Visočica limestone is extremely pure, with no visible metallic minerals, while Klikovača limestone contains Fe-mineral (most probably hydroxide due to the film-like appearance), but also Bi-bearing mineral (Fig. 3). However, the total amount of impurities is rather low as seen from geochemistry of stone (Tables 2 and 3) as well as XRD analyses (Fig. 4).

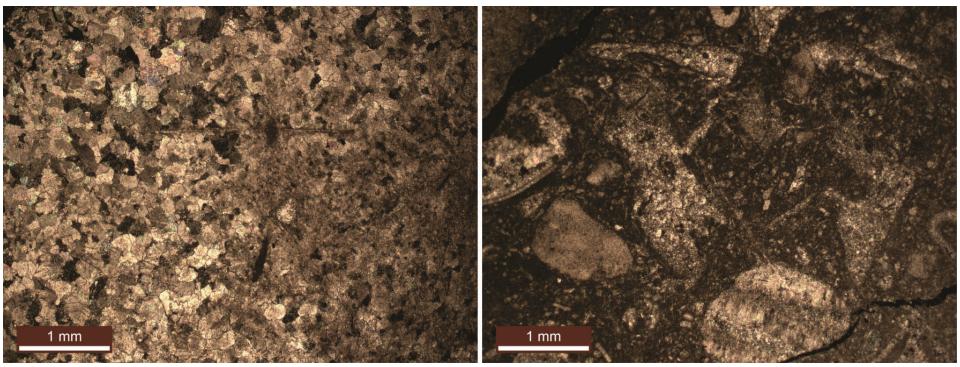
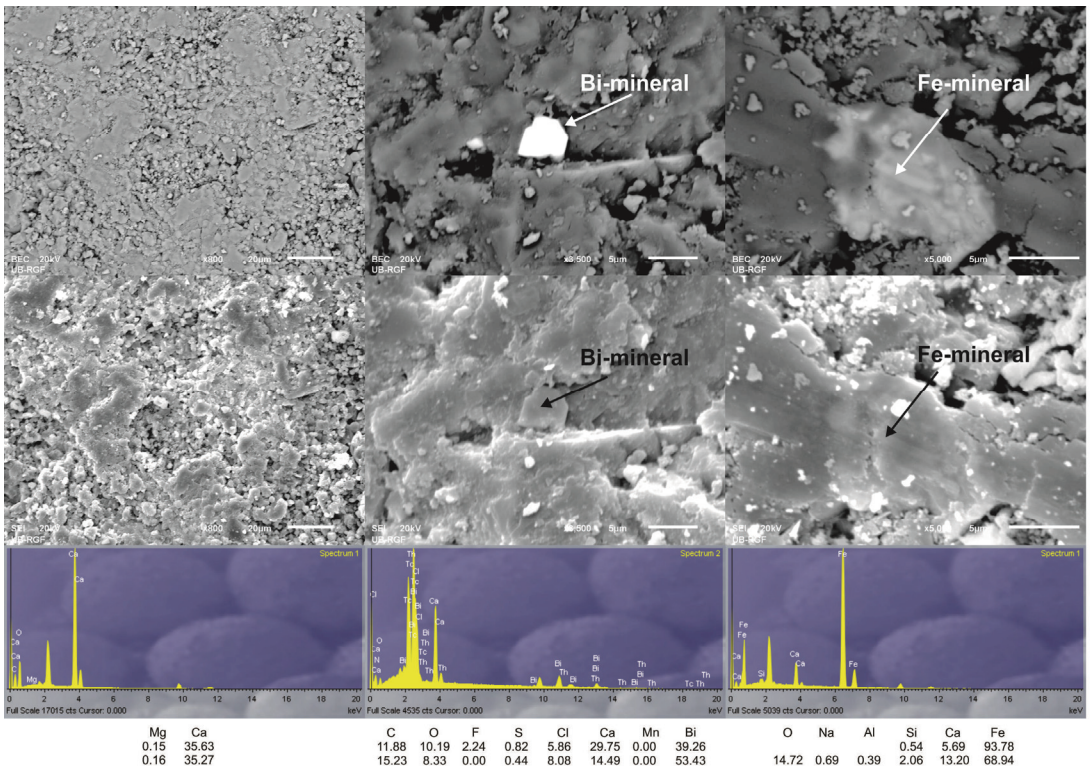


Figure 2. Photomicrographs of limestone from the Visočica deposit: grainstone (left image) and bioclastic packstone (right image).



Mg	Ca
0.15	35.63
0.16	35.27

C	O	F	S	Cl	Ca	Mn	Bi
11.88	10.19	2.24	0.82	5.86	29.75	0.00	39.26
15.23	8.33	0.00	0.44	8.08	14.49	0.00	53.43

O	Na	Al	Si	Ca	Fe
14.72	0.69	0.39	0.54	5.69	93.78
			2.06	13.20	68.94

Figure 3. SEM images of limestone from the Visočica (1) with none of the metallic minerals and Klikovača deposits (2 and 3) displaying metallic minerals; semiquantitative chemistry of analysed points is given below the images (in %).

Table 2. Chemical composition (in %) of limestone from the Bjelopavlići area

Site	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	Cr_2O_3	LOI	SUM	C_{total}	S_{total}
Suk	0.20	<0.01	0.04	0.06	55.84	0.34	<0.01	<0.01	<0.01	<0.01	<0.001	43.73	100.20	12.48	<0.02
Klikovača	0.10	<0.01	0.02	0.02	55.60	0.33	<0.01	<0.01	<0.01	<0.01	<0.001	43.76	99.84	12.26	<0.02
Sađavac	<0.10	<0.01	<0.01	0.03	54.84	0.67	<0.01	<0.01	<0.01	<0.01	0.003	43.87	99.47	12.28	0.03
Mali Garač	0.10	<0.01	0.03	0.02	55.04	0.70	<0.01	<0.01	<0.01	<0.01	0.002	43.88	99.79	12.44	0.03
Slatina	<0.10	<0.01	<0.01	<0.01	55.67	0.34	<0.01	<0.01	<0.01	<0.01	0.002	43.62	99.63	12.36	<0.02
Maljat	<0.10	<0.01	<0.01	0.03	55.58	0.27	<0.01	<0.01	<0.01	0.01	<0.001	43.27	99.22	12.48	<0.02
Vinici	<0.10	<0.01	<0.01	0.01	56.04	0.27	<0.01	<0.01	<0.01	<0.01	<0.001	43.39	99.70	12.39	<0.02
Visočica	<0.10	<0.01	0.01	0.02	56.13	0.28	<0.01	<0.01	<0.01	<0.01	0.003	43.40	99.86	12.36	<0.02
Pješivački do	<0.10	<0.01	<0.01	<0.01	55.27	0.56	<0.01	<0.01	<0.01	<0.01	0.003	43.67	99.52	12.47	0.02
Lalevići	0.10	<0.01	0.05	0.03	55.73	0.50	<0.01	<0.01	0.02	0.03	<0.001	43.76	100.20	12.26	0.02
Jovanovići	0.10	<0.01	0.03	0.02	55.43	0.53	<0.01	<0.01	0.01	0.03	0.001	43.72	99.89	12.39	0.02
Radujev krš	<0.10	<0.01	<0.01	0.01	55.84	0.28	<0.01	<0.01	<0.01	<0.01	0.006	43.57	99.72	12.22	<0.02

Table 3. Potentially toxic trace elements content (in mg/kg) in limestone from the Bjelopavlići area

Site	As	Be	Cd	Co	Cu	Hg	Mo	Ni	Pb	Se	Sb	Th	U
Suk	<0.5	<1	0.2	0.6	0.2	<0.01	<0.1	0.1	1.2	<0.5	0.1	<0.2	1.4
Klikovača	<0.5	<1	0.1	1.2	<0.1	<0.01	<0.1	0.2	1.1	<0.5	0.1	<0.2	0.7
Sađavac	<0.5	<1	0.2	0.4	0.1	<0.01	0.2	0.5	0.6	<0.5	<0.1	<0.2	1.3
Mali Garač	0.8	<1	0.2	0.6	0.1	<0.01	0.2	0.9	0.7	<0.5	<0.1	<0.2	1.4
Slatina	0.6	<1	0.4	0.5	<0.1	<0.01	<0.1	1.1	0.5	<0.5	<0.1	<0.2	0.9
Maljat	<0.5	<1	0.3	0.3	0.3	<0.01	<0.1	<0.1	0.2	<0.5	<0.1	<0.2	0.7
Vinici	<0.5	<1	0.2	0.8	<0.1	<0.01	<0.1	<0.1	0.2	<0.5	<0.1	<0.2	<0.1
Visočica	0.6	<1	0.2	0.7	0.1	<0.01	<0.1	0.8	0.1	<0.5	<0.1	<0.2	0.4
Pješivački do	<0.5	<1	0.3	0.2	<0.1	<0.01	<0.1	<0.1	0.2	<0.5	<0.1	<0.2	1.6
Lalevići	0.9	<1	0.2	1.0	0.3	0.02	<0.1	1.1	0.5	<0.5	<0.1	<0.2	1.2
Jovanovići	1.1	<1	0.3	0.7	0.2	0.01	<0.1	1.2	0.4	<0.5	<0.1	<0.2	1
Radujev krš	<0.5	<1	<0.1	<0.2	<0.1	<0.01	<0.1	0.7	0.1	<0.5	<0.1	<0.2	0.1

DTA and TGA, performed on all studied samples, confirmed that all limestones are very pure and, as the thermal curves were similar, only two are shown (Fig. 4).

Geochemical analyses (Table 2) revealed rather high purity of all studied limestone samples with $CaCO_3$ content of over 98 %, and small variations in MgO. The content of all other components is very low.

Potentially toxic trace elements (Table 3) show as well extremely low concentrations, particularly As, Cd, and Pb being below the most rigid requirements, enabling utilization of limestone in most demanding filler products like food industry [7] and pharmacy.

The basic parameters for using limestone as filler measured on investigated limestone are given in Table 4.

The current production of several products based on chemically pure limestone is from the Visočica quarry only, and filler is used for mechanical plaster, ceramic and marble adhesives, and glue for demit facades. By development of the chemical industry and the improvement of production technology, industry in Montenegro can significantly expand the possibilities to use carbonate fillers in various industrial branches: construction industry, paint and varnish industry, pharmaceutical and cosmetic industry, food industry, rubber industry, paper industry, industry of cables, polyvinyl chloride, and fertilisers. Very promising application of limestone filler in Montenegro is in cement and concrete industry. Namely, concrete with a high limestone filler content with suitable particle size distribution generally improves strength characteristics of concrete compared to the usual concrete with the same water/ /cement ratio and cement type [8–12]. The favourable opportunity is that quarry waste can be used for concrete mixtures [13]. That industry does not require extremely pure limestone, but the relatively cheap product.

As seen from the Table 5, limestone from several deposits can be used in practically all industrial branches without major limitations, while the other deposits can be used as a source of limestone for less demanding production. Further geological exploration and complete characterisation of limestone shall be conducted according to the existing regulations, but keeping in mind the potential usage of material. That should inc-

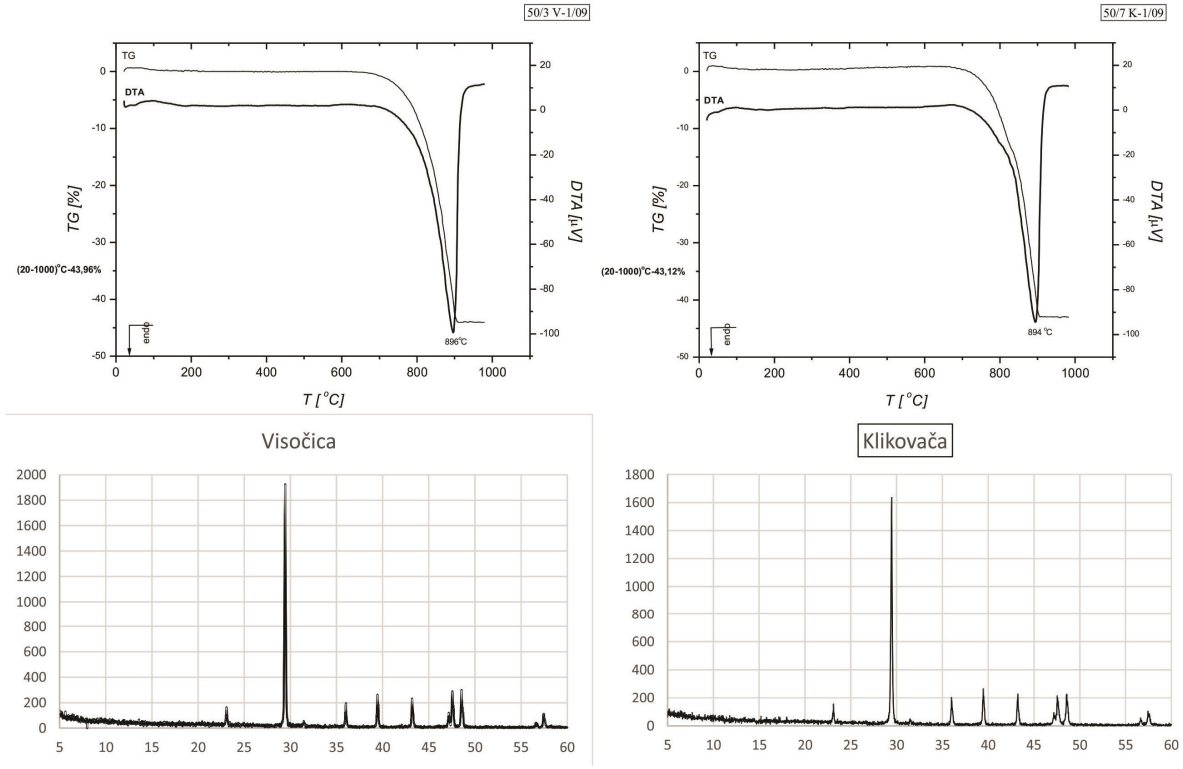


Figure 4. DTA, TGA and XRD curves of limestone from the Visočica and Klikovača deposits, displaying values for pure calcite.

Table 4. Basic technological parameters of limestone from the Bjelopavlići area

Deposit	Whiteness MgO-100%, %	Moisture %	Specific weight g/cm^3	Oil absorption %	Water absorption %	pH
Pješivački do	87.60	0.043	2.632	13.90	15.50	9.35
Radujev krš	93.50	0.051	2.726	12.00	15.50	9.20
Visočica	91.60	0.018	2.654	14.40	19.20	9.35
Suk	85.90	0.039	2.691	14.20	18.50	9.33
Sađavac	80.00	0.039	2.622	11.80	16.00	9.56
Slatina	91.90	0.046	2.689	14.60	19.50	9.34
Klikovača	84.90	0.064	2.654	13.90	19.00	9.39
Lalevići	84.50	0.022	2.632	11.80	15.50	9.33
Vinići	92.20	0.073	2.671	13.70	19.00	9.51
Maljat	93.40	0.087	2.681	13.00	19.20	9.40
Mali Garač	85.30	0.042	na.	11.80	15.80	na.
Jovanovići	85.80	0.045	2.679	11.80	15.00	9.35

lude much more analyses in order to define different grades of limestone that may be used in the most rational and sustainable way.

Limestone from the Bjelopavlići area as high quality raw material can be compared to other well known limestone resources in Serbia that can be used as coated filler in production of PVC products [14]. The importance of recycling dust from marble production, which would be economically rather important for the studied area, was discussed in recent years [15,16].

Although majority of papers dealing with detailed geochemistry of limestone is more geologically oriented [17–21], study in Canada [22] resulted in estimation of resources potential for industrial uses of limestone based on chemistry among the other criteria.

The overall economic potential of limestone fillers from the Bjelopavlići area is clearly visible from Table 6, where limestone resources are summarised. The most promising deposits for filler production are the actual dimension stone deposits, where the average block (and product) yield is around 20 %, while the rest is at

Table 5. Potential application of limestone from Bjelopavlići area based on preliminary tests. Y – yes, N – no, L – limited, VL – very limited

Deposit	Paints & varnishes	Paper	Rubber & PVC	Glass	Metal-lurgy	Foundry	Phar-macy	Sugar	Animal feed	Ferti-lizers	Soil neutralisation
Limitations		Whiteness		Chemistry			Whiteness			LOI	Chemistry P_2O_5, MgO, Cr
Radujev krš	Y	Y	Y	L	Y	Y	Y	Y	Y	Y	Y
Visočica	Y	Y	Y	L	Y	Y	Y	Y	Y	Y	Y
Slatina	Y	L	Y	L	Y	Y	Y	Y	Y	L	Y
Vinići	Y	Y	Y	L	Y	Y	Y	Y	Y	Y	Y
Maljat	Y	Y	Y	L	Y	Y	Y	Y	Y	Y	Y
Klikovača	Y	VL	Y	L	Y	Y	N	Y	Y	L	N
Lalevići	Y	VL	Y	L	Y	Y	N	Y	Y	L	L
Suk	Y	VL	Y	L	Y	Y	N	Y	Y	L	Y
Sađavac	Y	N	Y	L	Y	Y	N	Y	Y	L	N
Pješivački do	Y	VL	Y	L	Y	Y	N	Y	Y	L	Y
Mali Garač	Y	VL	Y	L	Y	Y	N	Y	Y	L	Y
Jovanovići	Y	VL	Y	L	Y	Y	N	Y	Y	L	N

Table 6. Resources of the stone and block mass of explored deposits; measured resources (000 m^3), JORC reporting

Deposit	Category (national reporting scheme)				Block yield	Block mass
	A	B	C_1	A+B+C_1	%	000 m^3
Suk	202	306	434	942	16.20	153
Klikovače	185	524	519	1228	25.00	307
Visočica	-	897	-	897	22.00	197
Maljat	376	635	868	1879	23.00	432
Slatina		234	291	525		
Jovanovići	251	592	1720	2563		
Vinići	348	347	645	1340	15.00	201
Radujev Krš	-	9	34	43	16.00	7
Sađavac		3285	1362	4647	Aggregate rock	

the moment not fully (or at all) utilised as either filler, or any other product.

In Montenegro the market for filler is rather low, but due to the relatively close sea harbour and direct railway connection as precondition for cheap transport, the possibilities for increasing the sales of this type of products in the future are open. One of the potential markets could be eventually Belgrade region in Serbia, which has no sources of high quality special aggregates on its territory [23]. However, the utilisation of studied limestone deposits will require a lot of planning at different levels and countries [24–26].

CONCLUSION

The first complex study of potential limestone resources for filler grade quality was performed on twelwe deposits in the Bjelopavlići area in Montenegro. The obtained results have proven the high quality of limestone from the studied deposits, as it can be used for paints and varnishes, rubber and plastics, metallurgy, foundry, sugar production, fertilizers and animal feed industry. Several deposits have very pure limestone, that can be also used for paper, glass, pharmaceutical industry and soil neutralisation. We would like to stress again that these are preliminary results based on representative samples from each studied deposit. Further exploration should prove the quality of different grades of limestone and shall also require more sensitive and precise analytical methods for Cr, P, F and some other components. The economic potential of limestone fillers from the Bjelopavlići area is high, and the most promising deposits for filler production are the actual dimension stone deposits, where the average block (and product) yield is around 20%, while the rest can be used for filler production.

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IZVOD

RESURSI KARBONATNIH PUNILA NA PODRUČJU BJELOPAVLIĆA, CRNA GORA

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Karbonatna punila predstavljaju veoma važnu mineralnu sirovinu koja se može upotrebiti u različitim industrijskim granama. Područje Bjelopavlića u Crnoj Gori odlikuje se prisustvom mineraloški i hemijski veoma čistih krečnjaka, koji su dosada istraživani uglavnom kao arhitektonsko-građevinski i tehnički građevinski kamen. Istraživani krečnjaci su senonske starosti, slojeviti do masivni, izgrađeni od različitih varijeteta – bioklastičnih sparita i mikrita, uglavnom sa rudistima i for a-miniferama. Uzorci za proučavanja (težine 80–100 kg) uzeti su iz svih istraživanih ležišta uzimajući u obzir makroskopski sastav stenske mase zbog reprezentativnosti. Zatim su iz njih izdvojeni manji komadi krečnjaka za potrebe izrade preparata za ispitivanja na optičkom i skenirajućem elektronskom mikroskopu. Ostatak uzoraka iz svakog ležšta je izdrobljen, homogenizovan i pulverizovan. Mineraloško–petrografska i tehnološka ispitivanja obuhvatila su optičku mikroskopiju, skenirajuću elektronsku mikroskopiju (SEM) i rendgensku difrakciju praha na dva izabrana uzorka iz ležišta Visočica i Klikovača, diferencijalno-termičke i termogravimetrijske analize (DTA i TGA), određivanje sadržaja vlage, beline, specifične težine, apsorpciju ulja i vode i određivanje pH. Geohemijska ispitivanja urađena su u AcmeLabs u Kanadi i to makro elementi XRF metodom (C i S Lecco metodom), a mikroelementi ICP-MS metodom. Istraživanja i ispitivanja dvanaest ležišta i pojava krečnjaka pokazala su visok kvalitet ove mineralne sirovine, pri čemu se krečnjak iz svih ležišta može upotrebiti u industriji boja i lakova, gume i plastike, metalurgiji, livarstvu, proizvodnji šećera, mineralnih đubriva i stočnoj hrani. Nekoliko ležišta i pojava sadrže veoma kvalitetan krečnjak koji se može upotrebiti i u industriji papira, stakla, farmaciji i neutralizaciji zemljišta. Potencijalni mineralni resursi procenjeni su na oko 23 miliona tona. U slučaju relativno jeftinog transporta postoji mogućnost snabdevanja i tržišta Srbije (pa i Beograda) agregatom za punila visokog kvaliteta.

Ključne reči: Krečnjak • Punila • Geohemija • Primena • Resursi