CERCETĂRI PRIVIND OBȚINEREA CLINCHERELOR DESTINATE REALIZĂRII CIMENTURILOR CU CAPACITATE DE ATENUARE A RADIAȚIILOR
RESEARCH REGARDING OBTAINING OF THE CLINKERS DESIGNED FOR PERFORMING CEMENTS WITH CAPACITY OF RADIATIONS ATTENUATION

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The paper presents the results of laboratory researches regarding the possibility of using, as addition in the raw mix for cement of barite – alternative raw material with high specific weight. The raw mixes with barite constitute the base for prepare of the clinkers designated to obtaining the cements with capacity of attenuation of gamma radiations. Technological behavior of the raw mixes with addition of barite was investigated in comparison with those of a raw mix obtained from current raw materials, considered as reference sample.

It was investigated the influence of barite on clinkers characteristics, from chemical, mineralogical and structural point of view, using X-ray diffraction, scanning electron microscopy (SEM analyses) .

From obtained clinkers, cements type CEM I in accordance with SR EN 197-1were performed. These cements were characterized both regarding to usual physical-mechanical properties and of the capacity for attenuation of gamma radiations. In order to evaluate the capacity of gamma radiations attenuation it was determined the dose rate equivalent for different radioactive source (Ir-192, Cs-137, Co-60). The presence of barite, in raw mixes composition for the clinkers, determined the increase of the value for linear coefficient of attenuation and an increased capacity of gamma radiations attenuation, respectively.

Lucrarea prezintă rezultatele cercetărilor de laborator privind posibilitatea utilizării, ca adaos în amestecul brut pentru ciment, a baritei – materie primă alternativă cu greutate specifică mare. Amestecurile brute cu barită au stat la baza realizării clincherelor destinate obținerii cimenturilor cu capacitate de atenuare a radiațiilor gamma. A fost urmărită comportarea tehnologică a amestecurilor brute cu adaos de barită, comparativ cu un amestec brut obținut din materii prime curente, considerat etalon.

S-a investigat influența baritei asupra caracteristicilor clincherelor, din punct de vedere al compoziției chimice, mineralogice şi structural – texturale, prin analize chimice, difractometrie cu raze X, microscopie electronică cu baleiaj (SEM)

Din clincherele obținute s-au realizat, conform SR EN 197-1, cimenturi tip CEM I care au fost caracterizate atât din punct de vedere fizico-mecanic cât şi al capacității de atenuare a radiațiilor. Pentru evaluarea capacității de atenuare a radiațiilor gamma s-a determinat debitul echivalentului de doză pentru diferite surse radioactive (Ir-192, Cs-137, Co-60). Prezența baritei, în rețeta compozițională a clincherelor, a determinat creşterea valorii coeficientului liniar de atenuare, respectiv o capacitate de atenuare mărită a radiațiilor gamma.

***Keywords**: Clinker, Barite, Cement, Linear coefficient of attenuation*

1. Introduction

Using of radioactive materials for electrical energy supply, in industry, medicine or for research lead inevitable to generation of radioactive wastes, for which administration the development of an integrated program, that include definitive storage is necessary. This program must fulfill fundamental objective of security, such as the protection of people health and of environment, now and in future, by undesired effects of ionizing radiations [1].

The storage of resulted radioactive wastes, in accordance with imposed conditions by UE, may be done only in distinct spaces, special arranged by environment protection [2]. This is a worldwide program because the danger on which these wastes represent, is determined by the period in which the wastes keep their radioactive properties, this may be extend on thousands of years. The decreasing of gamma radiations intensity is performed by using of some protection screens that determine the absorption and spreading of the radiations fascicle. Protection capacity is dependent both by the material nature from which the screen is made and also by its geometry [3-6].

In Romania, the most used protection method against radiations is represented by using

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of protection screens from lead or concrete. The use of these materials presents the disadvantage of necessity of performing some protection screens with considerable thicknesses.

Technological developments in the range of materials allowed performing of some new concretes with special properties. The cements used at obtainment of some concretes are special cements that must develop mechanical strengths similar to those used in the concretes of structure and in this category can be considerate aluminate cements, sulphate-aluminate cements, cements with sulphated slag, but especially cements with barium or with boron silicate, compounds with high specific weight, which act as good protectors against radiations [7].

For screening of gamma radiations, in present very hard concretes performed from usual cements (Portland cements without addition, Portland cements with additions of puzzolana, magnezia aluminate cements, some slag cements) and special aggregates are used [8-11]. These types of concrete have manifold uses especially in the domain of radioprotection against nuclear radiation. Among them we mention the following application areas: mean of protection in the construction of nuclear power plant, protection material for nuclear reactors, performing of protection walls in the atomic physics laboratories, protection of warehouses or rooms for exposure in which is worked with X-ray, gamma and neutron radiation sources, performing of anti-atomic shelters.Generally, very hard concretes are imposed as solutions only in the cases when, from technological reasons or from other causes, a strictly limit of the thickness for protection walls is required.

The paper presents the results of laboratory research performed in order to obtain Portland cements with attenuation capacity for gamma radiations. For obtain of such cements, clinkers resulted from the raw mixes containing barite – an alternative raw material with high specific weight.

2. Experiments

2.1. Materials

The raw materials used for obtainment of the raw mixtures were: limestone, marl, pyrite ash, sand and barite.

Chemical characteristics of used raw materials are presented in the Table 1.

In order to investigate the influence of barite addition on technological behavior of the raw mixture and on cement's screening capacity of gamma radiations, three raw mixture were performed: a standard mixture, from current raw materials (K1) and two mixtures in which besides current raw materials the barite in different proportions was added (K2, K3).

The calculations of dosage were made based on oxide composition presented in the Table 1, taking into account the following modular caracteristics: saturation degree S_k: 0.97; silica modulus M_{Si} = 2.5; alumina modulus M_{Al} = 1.6. The barite was dosed – in the raw mixtures, in proportion of 2% and 4%, respectively. The composition of the raw mixtures is presented in the Table 2.

2.2. Methods

For establishing of technological behavior, it was followed the influence of the barite presence on mixtures regarding grindability and clinker burnability, in comparison with the mixture of current raw materials.

From obtained clinkers were performed cements which were characterized from physical-mechanical point of view and of capacity for attenuation of the radiations.

Table 1

The chemical characteristics of the raw materials / *Caracteristicile chimice ale materiilor prime*

Raw material / *Materia primă*	Characteristic / *Caracteristica* (%)									
	PC	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	BaO	Ba SO_4
Limestone/ *Calcar*	43.60	54.22	0.36	1.12	0.39	0.39	0.02	0.04	nd	nd
Marl / *Marnă*	15.66	16.75	44.66	12.83	5.20	1.84	0.91	1.55	nd	nd
Sand/ *Nisip*	0.85	0.24	93.18	3.14	1.11	0.12	0.26	0.84	nd	nd
Pyrite ash/ *Cenuşă de pirită*	1.63	3.23	12.73	2.32	75.00	1.91	1.75	0.80	nd	nd
Barite / *Barită*	0.54	0.55	1.54	1.17	2.63	0.20	0.01	0.01	61.43	90.67

Table 2

The dosage of the raw mixtures / *Dozajul amestecurilor brute*

Specification/ *Specificația*	Raw mixture/*Amestec brut*		
	K 1	K 2	K 3
Raw materials dosage */ Dozaj materii prime (%)*			
Limestone / *Calcar*	73.63	73.63	73.63
Clay / *Argilă*	19.77	19.77	19.77
Pyrite ash / *Cenuşă pirită*	1.00	1.00	1.00
Sand / *Nisip*	5.59	5.59	5.59
Barite / *Barită*	-	2	4

The grindability was determined, by CEPROCIM method [12], in the laboratory mill with the diameter of 540 mm and the length of 560 mm. Loading with grinding media charge, for the first stage of grinding (coarse), was 76.90 kg balls, for final grinding (finishing) being performed with a loading of 144.3 kg bi-cones. For measuring of energy specific consumption, the mill was provided with an energy counter. Assessment of energetic specific consumption at industrial scale was performed for a residue on the 90 µm sieve, corresponding to 10 and 15%. In order to evaluate the energy specific consumption at industrial scale a correlation coefficient and data obtained in laboratory for grinding of raw mixes at reference fineness of 10% was used. In the case of clinker grindability determination, the reference fineness was corresponding to a specific surface area of 2500 cm^2/g. The assessment of energetic specific consumption at industrial scale was performed for two fineness, corresponding to specific surface areas of 3500cm^2/g and 4000 cm^2/g, respectively [12].

The raw mixtures grinded at fineness, corresponding to a residue on sieve of 90 µm (R_{009}), of about 10% were subjected to burning in a chamber kiln with gas at the maximum temperature of 1450°C, with maintaining of a plateau of 30 minutes, followed by fast cooling in air.

Clinker burnability was assessed by determination of free CaO content at different temperatures till 1450°C. Also in order to appreciate clinker burnability a series of specific technological indicators were calculated.

Obtained clinkers were characterized from chemical point of view. The influence of barite addition on mineralogical and structural characteristics was also investigated, by X-rays diffraction analyses and scanning electronic microscopy (SEM) coupled with well EDAX.

The cements were obtained by clinkers grinding together an addition of gypsum, in a laboratory mill, up to a corresponding fineness of a Blaine specific surface area of about 4000cm^2/g.

Compressive strength of the cements was determined on prisms of mortar, in accordance with SR EN 196-1 [13].

Testing of capacity of radiations screening was performed on hardened cement pastes, with water/cement ratio of 0.4. Obtained pastes were casted in parallelepiped matrix with the dimensions of 210 x 300 mm and thicknesses of 10, 20, 30, 50, 70 and 90 mm. The samples were kept, for 28 days in water at a temperature of 20°C. It was determined the dose equivalent rate (with flow-meter model Eberline FH-40-GL) for the following closed radioactive sources: Iridium-192 gamma energy average 0.37 MeV, Cesiu-137 gamma energy average 0.662 MeV and Cobalt-60, gamma energy average 1.25 MeV.

3. Results and discussions

3.1. Grindability of the raw mixtures

The results of tests regarding grindability of raw mixtures are presented in the Table 3.

It was stated that specific energy consumptions at grinding, industrial assessed, for the three mixtures are comparable, the addition of 4% barite (mixture K3) influencing the grinding in the way of a very slight increasing (~1%) of specific electric energy consumption in comparison with the reference (mixture K1).

3.2. Clinker burnability

Clinker burnability was evaluated by determination of free CaO content on smples thermalt treated at 1350°C, 1400°C, 1450°C mentaining a of 30 minutes. In order to obtain some supplementary data regarding the behavior at burning, the following indicators were calculated (based on chemical analysis of the clinkers) [14,15]:

- burnability indicator BI;
- burnability factor BF;
- burnability of clinker BC;
- quantity of liquid phase;
- minimum burning temperature for clinkerisation.

The results obtained for free CaO are presented in the Table 4.

Table 3

Grindability of raw mixtures / *Aptitudinea la măcinare a amestecurilor brute*

Raw mix *Amestec brut*	Specific electric energy consumptions, industrial assessed at / *Consum specific de energie electrică (kWh/t) estimat industrial la:*	
	R_{009} = 10%	R_{009} = 15%
K 1	26.74	22.03
K 2	26.73	22.04
K 3	26.91	22.19

Table 4

Variation of free CaO in clinkers versus temperature / *Variația CaO liber în clinchere funcție de temperatură*

Temperature / *Temperatura*(°C)	Free CaO/*CaO liber* (%)		
	K 1	K 2	K 3
1350	4.5	4.1	3.8
1400	2.3	1.9	1.7
1450 plateau beginning / *inceput palier*	1.4	1.1	0.8
1450 plateau ending / *sfârşit palier*	1.1	0.7	0.5

Table 5

Burnability indicators / *Indicatori de apreciere ai aptitudinii la clincherizare*

Indicator/*Indicator*	Allowable range *Interval admisibil*	Mixtures / *Amestecuri* (%)		
		K 1	K 2	K 3
Burnability indicator BI / *Indicele aptitudinii la ardere BI*	2.6 – 4.2	3.73	3.66	3.61
Burnability factor BF / *Factorul de aptitudine la ardere BF*	100 – 120	118.90	116.83	115.64
Burnability of clinker BC / *Capacitatea de clincherizare BC*	-	32.96	30.26	28.93
Liquid phase (%) / *Faza lichidă (%)*	23 – 28	22.74	23.51	23.63
Minimum burning temperature (^oC) / *Temperatura minimă de ardere (^oC)*	-	1447	1442	1437

The presence of barite in the raw mixture determines a better behavior at clinkerization, values of free CaO below 2% (maximum value technological recommended) being obtained for the temperature of 1400°C. The barite has the role of mineralizator in the raw mixture clinkerization process. It could be supposed an increase of the liquid phase proportion, a decrease of its viscosity, favoring the processes of diffusion and consequently CaO binding, with formation of the mineral compounds [16,17].

Calculated values of the indicators that characterize clinker are presented in the Table 5.

It is remarked the improving of clinker burnability in the same time with increasing of proportion of barite from the raw mix.

Values of indicators that characterize clinker burnability are correlated with the values of free CaO content, confirming this way that the barite presence in the raw mix lead to a better burnability in comparison with standard mixture.

3.3. Chemical and mineralogical characteristics of the clinker

Chemical and potential mineralogical composition (calculated with Bogue's relations), and also the modular characteristics for the three clinkers are presented in the Tables 6 and 7.

Table 6

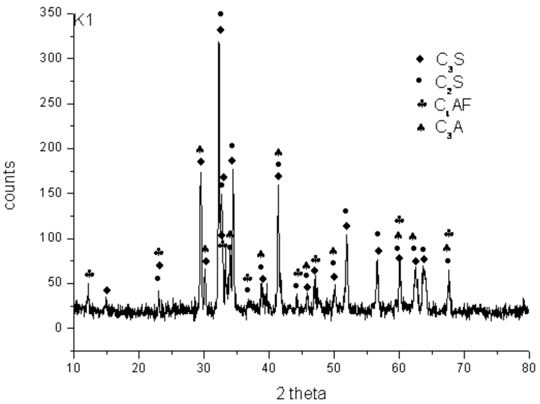
The chemical composition of the clinkers / *Analiza chimică a clincherelor*

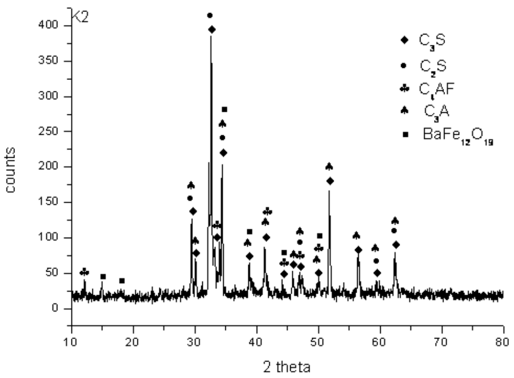
Sample/ *Proba*	Characteristics / *Caracteristica* (%)									
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	Na_2O	K_2O	SO_3	CaO_{liber}	BaO
K 1	67.16	21.66	5.12	3.28	1.15	0.09	0.02	0.32	1.18	0.0
K 2	66.87	21.67	5.22	3.49	1.21	0.09	0.03	0.41	0.65	1.87
K 3	67.54	22.09	5.20	3.57	1.9	0.08	0.03	0.61	0.43	3.62

Table 7

The modular characteristics and mineralogical composition of clinkers / *Compoziţia mineralogică şi modulară a clincherelor*

Sample/ *Proba*	S_k	M_{Si}	M_{Al}	C_3S	C_2S	C_3A	C_4AF
K 1	0.97	2.54	1.56	67.21	11.79	8.02	9.97
K 2	0.98	2.44	1.50	67.83	11.10	7.93	10.61
K 3	0.97	2.49	1.46	67.22	13.18	7.75	10.85





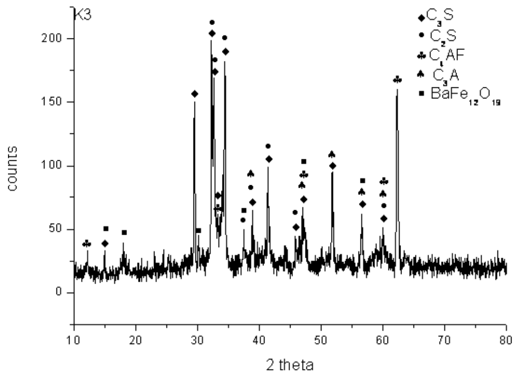


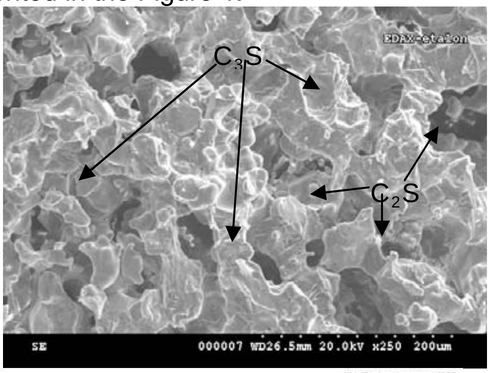
Fig. 1 – X-ray patterns of the clinkers
Difractograme ale clincherelor.

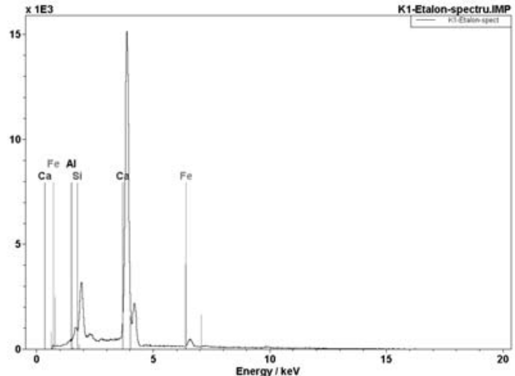
In case of the clinkers K 2 and K 3, resulted from the raw mixescontaining 2% and 4% barite, respectively, the content of free CaO is lower in comparison with that of reference clinker. It may be concluded the fact that in in parallel with the increase of barite proportion in the raw mixture, the process of clinkerization is favored. Modulus values are closed for all clinkers, being framed in proposed limits.

Mineralogical composition of the clinkers is also suggested by the diffraction spectrum, presented in the Figure 1.

X-rays diagram corresponding to the reference clinker K1 shows the existence of the compounds characteristic to Portland clinker: C_3S, C_2S, C_3A and C_4AF. In case of the clinkers K 2 and K 3, it can be seen the presence of some peaks corresponding to hexaferite of barium ($BaFe_{12}O_{19}$).

SEM images and EDAX spectrum corresponding to the clinkers are presented in the Figures 2-4.





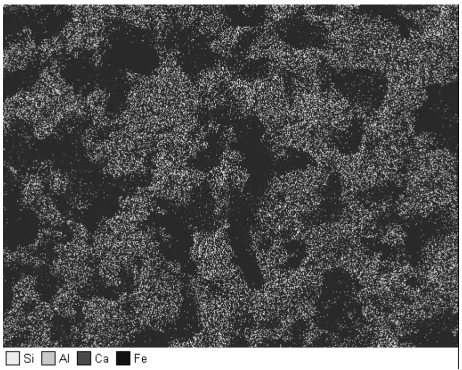
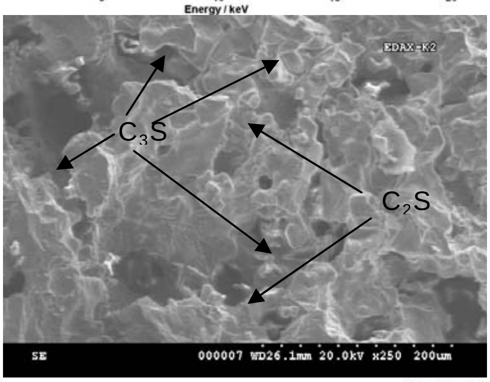
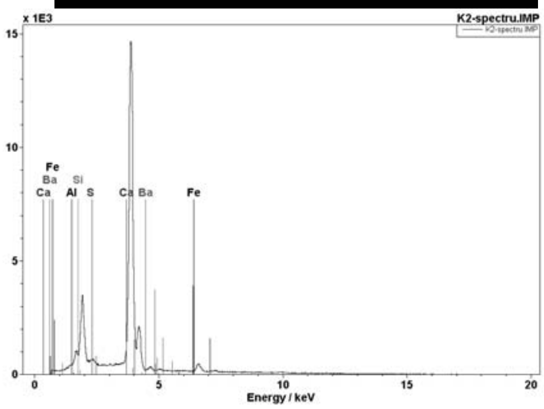


Fig. 2 - SEM images and EDAX spectrum for clinker K1
Imagini SEM şi spectru EDAX pentru clincherul K1.





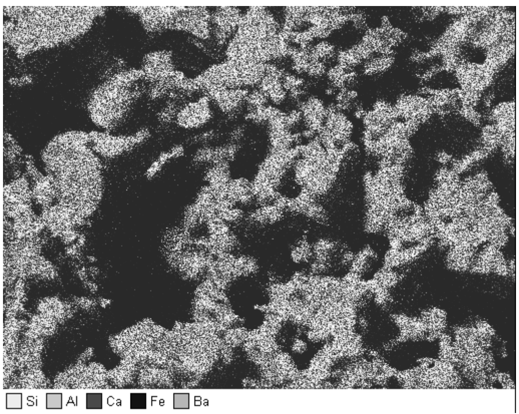


Fig. 3 - SEM images and EDAX spectrum for clinker K2
Imagini SEM şi spectru EDAX pentru clincherul K2.

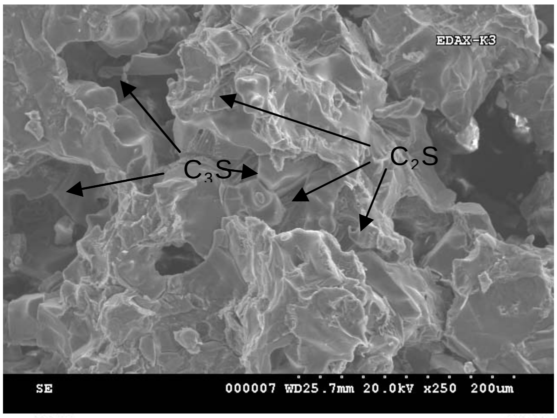
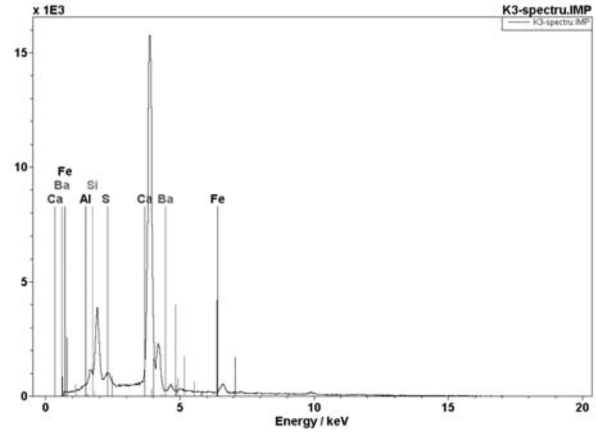
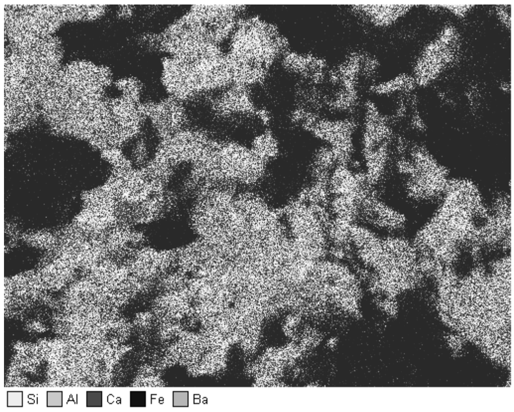


Fig. 4 - SEM images and EDAX spectrum for clinker K3
Imagini SEM si spectru EDAX pentru clincherul K3.





From the scanning electronic microscopy images characteristic to the clinkers K1 – K3, it can be remarked:

- the microstructure of the clinkers are characterized by a high porosity;
- presence of polyhedral formations characteristic to alite (tricalcium silicate - C_3S), round granules characteristic to belite (dicalcium silicate - C_2S) and acicular formations characteristic to celite (tetracalcium feritaluminate - C_4AF) besides vitreous interstitial phase;
- addition of barite determines some modifications in the phase and morphological characteristics of the clinkers – formation of alite more developed and a high quantity of vitreous phase; this influence is accentuated with the increasing of barite proportion.

EDAX spectrum and the images of elements distribution for analyzed clinkers put in evidence the presence of Ba which is evenly distributed in their mass.

3.4. Grindability of clinkers

The results of tests concerning grindability of clinkers are presented in the Table 8.

It is stated that the addition of barite in the raw mixture determines a slight increase of energy specific consumption at clinkers grinding, in comparison with the reference (K1). For 2% barite in the raw mixture the increase of specific energy consumption at grinding is about 2,0%, while the increase of barite proportion to 4% lead to an energy consumption at grinding increased with 3,5%.

3.5. Physical-mechanical characteristics of cements

Using the three clinkers, three cements – C1, C2 and C3 were prepared by grinding with an addition of 5% gypsum.

The main physical-mechanical characteristics of the cements are presented in the Table 9.

Table 8

Grindability of clinkers / *Aptitudinea la măcinare a clincherelor*

Clinker/*Clincher*	Electrical energy specific consumption (kWh/t) industrial assessed at / *Consumul specific de energie electrică estimat industrial la*	
	S_{Sp} = 3500cm^2/g	S_{Sp} = 4000cm^2/g
K 1	38.01	46.25
K 2	38.85	47.22
K 3	39.34	47.93

Table 9

Physical- mechanical characteristics of cements / *Caracteristicile fizico-mecanice ale cimenturilor*

Physical - mechanical characteristics/ *Caracteristici fizico-mecanice*		Cement symbol *Simbol ciment* C 1	C 2	C 3	Conditions imposed by/ *Condiții impuse de* SR EN 197-1
Water for standard consistency, % *Apa de consist. standard*		28.0	27.6	27.0	-
Setting time *Timp de priză*	Beginning / *Început, min*	160	170	190	≥ 60
	End / *Sfârşit, hours-min*	3-30	3-45	4-15	-
Stability / *Stabilitate, mm*		0,0	0,0	0,0	≤ 10
Compressive strength/ *Rezistența mecanică la compresiune MPa*	2 days / *zile*	26.9	25.5	24.1	≥ 20
	7 days / *zile*	38.6	38.1	37.2	
	28 days / *zile*	46.3	45.8	44.7	≥ 42.5 ... ≤ 62.5
Strength class / *Clasa de rezistență*		**42.5R**	**42.5R**	**42.5R**	**42.5R**

In case of the cements resulted from the raw mixtures with addition of barite, a slow increasing of the setting time can be remark, in correlation with increase of SO_3 content in the clinker.

Determined physical-mechanical characteristics are in accordance with the requirements imposed by SR EN 197-1 [18]. Mechanical strengths developed at the terms of 2 and 28 days allow framing of the cements C 2 and C 3 in the same class of strength, 42.5R, with that of the reference C 1.

3.6. Capacity of screening

It was determined the dose equivalent rate as the average of 20 individual measurements for each type of cement plate. The obtained values are represented in the Figure 5.

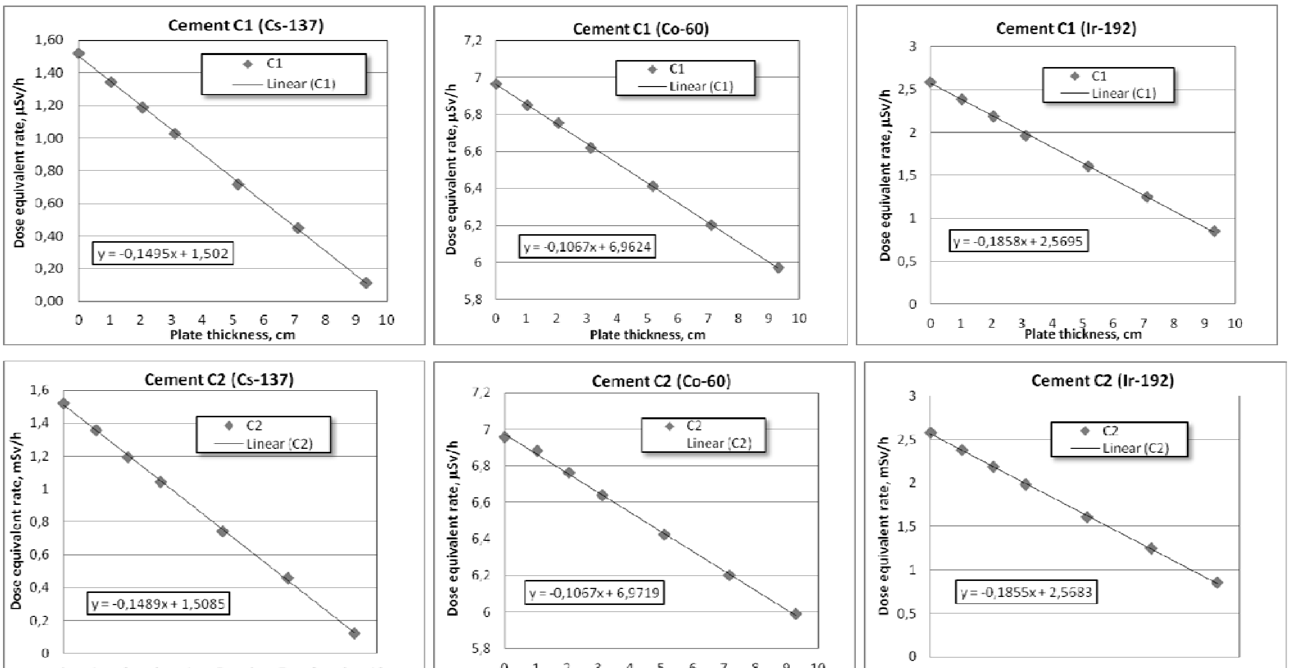


Fig. 5- Dose equivalent rate versus plate thickness / *Debitul echivalentului de doză funcție de grosimea plăcii* .

Table 10

Linear coefficient of attenuation / *Coeficientul liniar de atenuare*

Cement Plate/*Placa Ciment*	Ir-192	Cs-137	Co-60
Energy gamma radiation/ *Energie radiatie gamma*	0.370 MeV	0.662 MeV	1.250 MeV
Linear coefficient of attenuation, μ (cm^{-1}) / *Coeficient liniar de atenuare,* μ (cm^{-1})			
C 1	0.1222	0.1682	0.2056
C 2	0.1275	0.1732	0.2124
C 3	0.1286	0.1766	0.2162

From the line slope was determined the value of linear attenuation coefficient for each type of plate, for each energy of the fascicle of gamma radiation [19-21].

The values obtained for linear coefficient of attenuation are presented in the Table 10.

Linear coefficient of attenuation for the cement resulted from the mixture containing 4% barite show an increase of about 4% in case of the source of Ir-192, about 5% for the sources of Cs-137 and Co-60, in comparison with standard cement, respectively.

4. Conclusions

The obtained laboratory clinkers that used barite added in proportion of 2% and 4% in raw materials, were further used for development of cements with the capacity of radiation attenuation. The following conclusions can be drawn up from our laboratory studies:

- from technological behavior point of view, use of barite in the raw mixture leads to an increase with about 1% of energetic specific consumption and an improvement of clinker burnability;
- microstructure of the clinkers with barium content are characterized by a higher porosity, the presence of more developed crystals of alite and a higher quantity of vitreous phase;
- the presence of barite in the raw mixture leads to an increase of energy consumption at clinker grinding; the highest increase of consumption, of about 3.5%, was determinated for the clinker resulted from the raw mixture with 4% barite;
- the compressive strengths values allow the inclusion of all the cements in the strength class 42.5R;
- the presence of barite, in proportion of 4% in the raw mixture leads to an improvement of the screening capacity for gamma radiations with 4-5%, depending on the used type of radioactive source.

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