UNIVERSAL WISER
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Research Article

Effect of Boron Minerals Colemanite and Ulexite on Physical, Chemical and Mechanical Properties of Cement

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Abstract: This study investigated how the boron minerals colemanite and ulexite affect the physical, chemical and mechanical properties of cement. The radioactive permeability of the mortars with added colemanite and ulexite is also studied. The initial and final setting times, volume stability, and compressive and flexural strengths are determined. Chemical and micro-structural examinations are also performed on the cements. The linear attenuation coefficients of the mortars added to colemanite and ulexite are measured. It is concluded that colemanite and ulexite can be used as an additive in the production of cement to delay initial setting time and to reduce expansion. Also, it has been determined that colemanite reduces the hydration heat of cement. The linear attenuation coefficients of the mortars added colemanite and ulexite are also increased.

Keywords: colemanite, ulexite, farina, initial setting time, volume stability

1. Introduction

Turkey has rich boron mines, operates boron mines and produces different boron mineral derivatives. During the operation of the boron mines, approximately 250 million tons of waste are generated annually, mainly boron-containing mud [1]. There are many scientific studies which examine how to utilise the boron mine waste and prevent damage to the environment. Among these studies, the utilisation of the wastes of boron minerals in the cement and concrete industry holds an important place [1-7].

The effect of the boron minerals on the cement properties was studied by researchers [1, 8-11], and they found that boron minerals extend the initial setting time of cement. Abali et al. [1] studied the boron mineral with tincal included B_2O_3 in the cement paste mixture. It is determined that the lower amount of B_2O_3 (< 0.2%) does not have much effect on the initial setting time, but the higher amount of B_2O_3 (> 0.6%) increases the initial setting time of the cement. It is observed that the cement included tincal up to 2% of its mass, increasing the initial setting time of the cement to more than ten hours. Targan et al. [8] concluded that the maximum rate of colemanite is 4% in the pozzolanic cement. Ozdemir and Nese [12] used boron-containing clay waste in cement and determined that mud waste containing boron between 5% and 10% can be used in cement production.

High strength is one of the most essential properties of cement, but using boron mineral or its waste decreases the compressive strength of the cement [1, 8, 13]. In some studies, it is stated that a small amount of boron waste, such as 1% of the cement waste, increases the compressive strength of the cement [8, 12]. This is due to the low amount of B_2O_3 in the waste and the heterogeneous structure of mortar samples. There is no evidence in the other studies of cement

containing boron wastes that the boron mineral increases the compressive strength of cement.

Some researchers [2, 4, 9, 13] studied cement including boron gypsum instead of gypsum. Elbeyli et al. [9] determined that the cement with boron gypsum decreases the expansion of the cement and has a positive effect on cement mechanical properties. However, some researchers [2, 4, 14] stated that when boron with gypsum is used instead of as gypsum, it negatively affects cement, increasing the initial setting time.

Some studies examined the effect of boron minerals on the radioactive permeability of concrete [1, 9, 11, 14]. It is stated in these studies that because boron has a small atomic number, it can be beneficial for neutron shielding [15-16]. Kharita et al. [11] found that boric acid or borax is unsuitable for cement because they increase the initial setting time and, in the case of using them, maximum at 0.5% of cement mass. There is no clear effect on neutron shielding, but it can effectively attenuate gamma rays. Oto et al. [17] determined that the gamma-ray attenuation of concrete with colemanite is better than concrete without colemanite. However, very little difference was detected in concrete with colemanite (from 2.5% to 20%) by considering the gamma-ray attenuation [18].

Although there are some studies related to the effect of boron-containing waste minerals on the physical, chemical and mechanical properties of cement, there needs to be a detailed study on the effect of colemanite and ulexite on the hydration heat of cement. Also, limited studies were performed examining the radioactive permeability of mortars produced with colemanite and ulexite. Under these considerations the main target of this study is to contribute to the literature by performing a detailed studies relating with these lacks. In this scope, the initial and final setting time, the volume stability, compressive-flexural strength, hydration heat and linear attenuation coefficients are measured in three different types of cement containing colemanite and ulexite. Depending on the experimental data, the effect of colemanite and ulexite mixed in cement with three methods, i) directly with portland cement, ii) while clinker grained, and iii) while farina baked, on the mechanical and physical properties of cements are determined and evaluated.

2. Materials and methods
2.1 *Materials*

In this study, three types of cements were used. First, the cement (Cement-A) is prepared by mixing the CEM I 42.5 R cement with colemanite and ulexite by specific proportions (0.5%, 1%, 2%, 3%, 4%, and 5%). The second type of cement (Cement-B) is produced by grinding the clinker with gypsum and boron minerals in the laboratory with a ball mill. The third type of cement (Cement-C) is obtained by grinding the clinker with gypsums in the laboratory, which is produced by bucking the farina obtained at high temperatures. The chemical composition of Cement-A, Cement-B and Cement-C are given in Table 1.

The boron minerals are obtained from the facility of Eti Maden General Directorate in Bigadiç, Balıkesir. Colemanite and ulexite are used in the study as boron minerals. The ground colemanite and ulexite minerals are added to the cement. The chemical compositions of boron minerals are shown in Table 1.

Table 1. Chemical composition of the cements and Boron minerals. colemanite and ulexite (%)

Chemical compounds	Cement-A	Cement-B		Cement-C			Colemanite	Ulexite
		Clinker	Gypsum	C-1	C-2	C-3		
CaO	61.47	64.17	-	60.95	60.35	60.94	26.59	15.70
SiO_2	19.92	20.46	-	22.77	23.24	22.84	5.56	3.31
Al_2O_3	7.01	5.07	-	6.37	7.65	6.97	0.17	0.03
Fe_2O_3	2.95	3.47	-	4.19	4.47	4.52	0.032	0.027
MgO	1.12	1.35	-	0.64	0	0	2.81	1.31
SO_3	2.51	0.83	43.6	1.44	1.10	0.73	0.24	0.04

Table 1. (cont.)

Chemical compounds	Cement-A	Cement-B		Cement-C			Colemanite	Ulexite
		Clinker	Gypsum	C-1	C-2	C-3		
Na_2O	0.34	0.25	-	0.14	0.11	0.13	0.15	3.81
K_2O	1.11	0.50	-	0.18	0.13	0.16	-	-
Cl^-	0.008	0.006	-	0.022	0.022	0.002	-	-
Loss on ignition	2.52	0.50	-	2.68	1.96	2.54		
Total	98.95	96.6	-	98.63	99.03	98.83		
Undetermined	1.05	3.39	56.3	1.37	0.97	1.17		
Cristal water	-	-	16.7	-	-	-		
Free lime	1.16	1.54	-	3.96	3.58	2.34		
Insoluble residue	0.94	0.56	-	2.07	3.96	3.14		
Total contribution	0	0	-	0	3.32	4.36		
B_2O_3							39.8	37.11
As_2O_3							32	59.79
As (ppm)							24.24	45.30
SrO							1.19	0.82

2.2 *The experiments method*

The preparation of mortar samples and the analysis of flexural and compressive strength are performed according to TS EN 196-1 [18]. However, the standard consistency, the initial setting time, the final setting time and volume stability analyses of the cements are done according to TS EN 196-3 [19]. The sieve and chemical analyses of cements are performed according to TS EN 196-6 [20] and TS EN 196-2 [21], respectively. Scanning electron microscopes are used for micro-structural analysis of the hardened ulexite and colemanite cement pastes. Samples used for experiments are obtained by cutting the hardened cement paste with dimensions of 6x6x6 mm. They are dried using an oven, coated with gold in a vacuumed condition, and examined by a tunnelling scanning microscope.

The hydration heat of cement containing colemanite is measured by dissolving the dry cement and hydrated cement in a solution including 40%, 2,760 *grams* of hydrofluoric acid into each of the 2 ± 0.01 *mole/liter*. 100 *gram* nitric acid solution according to TS EN 196-8 [22]. The ratio of the total acid mixture mass to the dry cement mass is mixed as 140 ± 2, and the increment in temperature of the solution is measured by 0.002 °C precision of the Beckmann thermometer. After seven days hardened cement paste, which is prepared by adding 100 *gram* of cement into 40 *gram* of water, is ground and sieved. The hydrated cement, which has 40% more mass than the dry cement, is dissolved in the acid, and the Beckmann thermometer measures the temperature of the solution. The hydration heat of the cement is calculated by using the formulas given in Eq. (1).

$$H_i = Q_a - Q_i \tag{1}$$

where Q_a is the heat of dissolution of the dry cement in the solution. Q_a is calculated by using Eq. (2).

$$Q_a = \left(C.\Delta T_c / P\right) + 0.8\left(T_f - T_a\right) + 0.8(Tf - 20) \text{ (Unit: } J / g) \tag{2}$$

where ΔT_c is the corrected temperature increase (*inK*), C is the thermal capacitance of the calorimeter (*J/K*), P is the amount of dry cement sample (*gram*), T_f is the temperature of the dry cement at the end of the dissolving period (°C), T_a is the solution temperature in the calorimeter just before the dry cement isn't added (°C). 0.8 is the specific heat of the dry cement (*J/g.K*) and, −0.8 is the temperature coefficient of the heat of dissolution of the dry cement (*J/g.K*).

ΔT_c is calculated by using Eqs. (3 and 4) for all the cements and Portland cement, respectively.

$$\Delta T_c = (T_{30} - T_0) - 2\left[(T_0 - T_{-15}) - K(T_{30} - T_0)\right] \tag{3}$$

$$\Delta T_c = (T_{30} - T_0) - 2(T_{45} - T_{30}) \tag{4}$$

where T_{30}, T_0, T_{-15} and T_{45} are the solution temperatures measured by Beckmann thermometer at $t = 30, 0, -15$ and 45 minutes, respectively.

Q_i is the heat of dissolution of the hydrate cement in the solvent and it is obtained by using Eq. (5).

$$Q_i = (C.\Delta T_c / P.F) + 1.7(T_f - T_a) + 1.3(T_f - 20) \text{ (Unit: } J/g) \tag{5}$$

where P is the amount of the hydrate cement sample (*gram*), F is the correction factor for bound water, 1.7 is the specific heat of hydrated cement (J/gK^{-1}) and −1.3 is the temperature coefficient of the heat of dissolved hydrated cement. The correction factor for bound water (F) is calculated by using Eq. (6).

$$F = (100 - m_h) / (100 - m_a) \tag{6}$$

where m_h is the mass loss of the hydrated sample after ignition (%) and m_a is the mass loss of the dry sample after ignition (%).

The gamma ray attenuations in the mortar samples for Cement-A are measured by the gamma ray detector are given in Table 2. The gamma ray attenuation of mortars with boron mineral are measured by Cesium source which has 661.6 *keV* energy and the gamma ray attenuation coefficient are calculated by the Lambert Beer law. The Canberra Marka GC1018 model HPGe detector, which has 1a 0% relative yield and 1.8 *keV* separation power at 1,332 *keV* for the ^{60}Co source is used for measurements.

Table 2. The amount of the materials used in the mortar samples (in grams)

	Sample name	Cement	Water	Colemanite	Ulexite	Standard sand
	HO	450	225	0	0	1,350
	K0.5	447.75	225	2.25	0	1,350
	K1	445.5	225	4.5	0	1,350
	K2	441	225	9	0	1,350
	K3	436.5	225	13.5	0	1,350
Cement-A	K4	432	225	18	0	1,350
	K5	427.5	225	22.5	0	1,350
	U0.5	447.75	225	0	2.25	1,350
	U1	445.5	225	0	4.5	1,350
	U2	441	225	0	9	1,350

Table 2. (cont.)

Sample name		Cement	Water	Colemanite	Ulexite	Standard sand
Cement-A	U2	441	225	0	9	1,350
	U3	436.5	225	0	13.5	1,350
	U4	432	225	0	18	1,350
	U5	427.5	225	0	22.5	1,350
Cement-B	N1	450	225	0	0	1,350
	K0.5	450	225	(% 0.5)	0	1,350
	K1	450	225	(% 1)	0	1,350
	K2	450	225	(% 2)	0	1,350
	K3	450	225	(% 3)	0	1,350
	U0.5	450	225	0	(% 0.5)	1,350
	U1	450	225	0	(% 1)	1,350
	U2	450	225	0	(% 2)	1,350
	U3	450	225	0	(% 3)	1,350
	SK	450	225	(% 3)	0	1,350
Cement-C	N	450	225	0	0	1,350
	% 1K	450	225	(% 1)	0	1,350
	% 2K	450	225	(% 2)	0	1,350

If a material of thickness x is placed in the path of a beam of gamma radiations, the intensity of the beam will be attenuated according to Beer-Lambert's law [23]

$$I = I_0 e^{-\mu x} \tag{7}$$

where I_0 and I are the unattenuated and attenuated photon intensities, respectively and μ (cm^{-1}) is the linear attenuation coefficient of the material.

2.3 *Producing of mortar samples*

Table 2 shows the amount of materials added to the mortar samples, HO, K and U in Cement-A, prepared by reducing the 450 *gram* cement mass in standard sample by 0.5%, 1%, 2%, 3%, 4% and 5% and replacing colemanite and ulexite for the missing cement mass to show the standard mortar with colemanite and ulexite samples, respectively.

The clinker amount was constant during the grinding of Cement-B for all types (see Table 2). N1 type for Cement-B is prepared by grinding clinker with 4% of gypsum. The other kinds of Cement-B are produced by adding colemanite and ulexite at 0.5%, 1%, 2% and 3% of the clinker amount while keeping the total boron mineral and gypsum at 4%.

Cement-C was produced by grinding gypsum and clinker, which was obtained by adding colemanite with 1% and 2% of marine mass into farina and farina with colemanite was baked in the stove at high temperature. Cement-C was produced in three types. The first was prepared by grinding the 4% gypsum and clinker without boron. The second one was produced by grinding clinker with 1% colemanite and 3% gypsum. The third one was obtained by grinding clinker

containing 2% colemanite and 2% gypsum. The colemanite and ulexite is added into Cement-B and Cement-C during the production period, thus the amounts of the boron added into the cement is given as percentage (%) in Table 2.

3. Results and discussion

The effect of boron minerals, colemanite and ulexite on physical chemical and mechanical properties of cement are presented in this study. Figure 1(a) shows the variation of initial setting time with colemanite for cement A, B and C. The initial setting time highly increases almost linearly with the colemanite rate for Cement-A and C. However, a similar variation was obtained for cement B for the colemanite rate $\leq 1\%$. Also, the variation of initial setting time depends on the ulexite rate given in Figure 1(b). It is observed that the initial setting time for both cement A and B significantly increases with the ulexite rate.
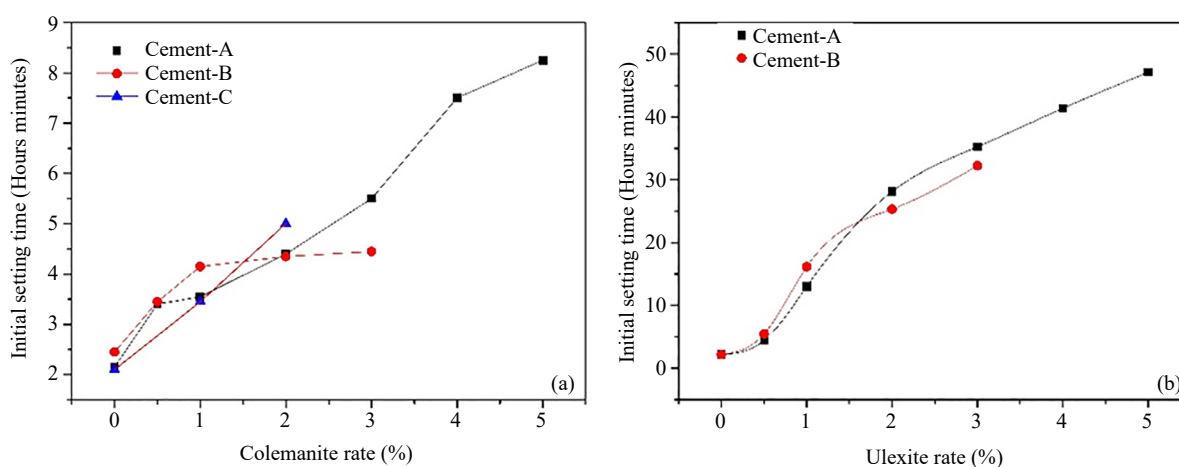
Figure 1. Initial setting times of cements, containing colemanite and ulexite

As seen in Figure 1 (a) and (b), the ulexite added to the cement significantly increases the initial setting time when considering the colemanite included in the cement. There was an excessive increase in the initial setting times of Cement-A containing colemanite and ulexite. In order to prevent this problem, Cement-B's were produced by reducing the amount of gypsum in the laboratory. As colemanite and ulexite are added into Cement-A, more that 3% of the setting problems occur in the cement. Even when colemanite is added into cement at very small amounts, such as 0.5%, there is a significant increase in the initial setting times of the cement. In the studies, the boron-containing cement, with a B_2O_3 ratio more than 0.2%, the initial setting times extend. Thus the 2.5% of B_2O_3 ratio can be used in the cement [1]. The ratios of B_2O_3 in the colemanite and ulexite are very close to each other and their values are 39.8% and 37.11, respectively (see Table 1). The chemical formula of colemanite is $2CaO \cdot 3B_2O_3 \cdot 5H_2O$ and the chemical formula of ulexide is $NaO_2 \cdot Ca0 \cdot 5B_2O_3 \cdot 12H_2O$. The different chemical element in both minerals is the Na^{+1} element. It is known that the initial setting time is increased by SO_3. When the chemical compounds of colemanite and ulexite are analysed in Table 1 ulexite increased the initial setting time more than colemanite, even though it does not contain SO_3, causing the increase in the initial setting time. The most obvious difference between the chemical compounds of colemanite and ulexite in Table 1 is that the Ca^{+2} element in colemanite is more than the one in ulexite and the amount in ulexite is more than the one in colemanite.
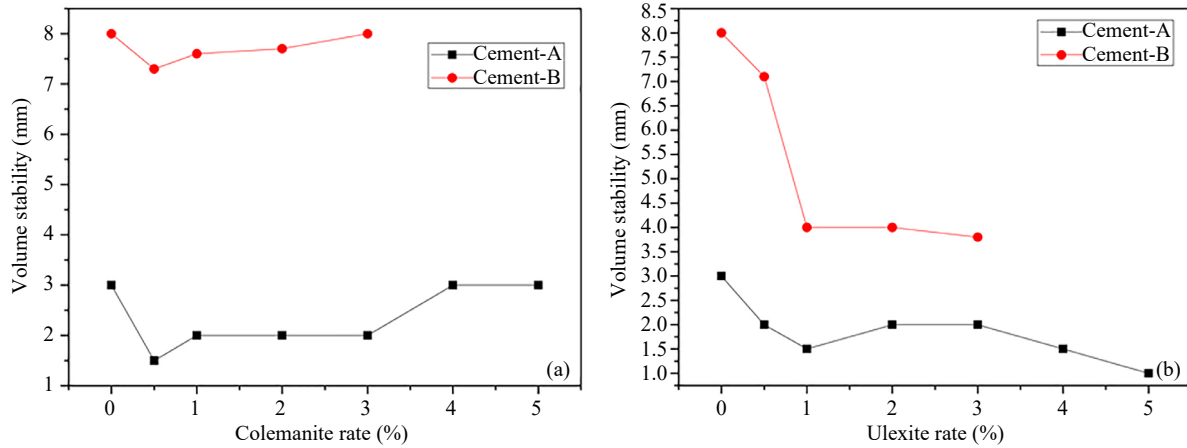
Figure 2. Variation of volume stability with (a) colemanite rate (b) ulexite rate

Figures 2 (a) and (b) present the variation of volume stability depending on the colemanite rate and ulexite rate, respectively. It is observed that the effect of the colemanite rate on the volume stability is very small, but volume stability highly decreases with the ulexite rate.

Figure 3 shows the 2-, 7-, 28- and 90-day changes of the compressive strength in the mortars with added colemanite named as Cement-A. The compressive strength of the standard mortar samples without boron mineral (*HO*) are higher than the compressive strength of the mortar samples with added colemanite. Thus, the compressive strength decreases when the amount of the cement goes down and the amount of colemanite increases in the mortar samples. This result shows that the colemanite has no pozzolanic effect. Also, the decreasing rate of the two-day compressive strength of mortars with colemanite is higher than the decreasing rate of the 90-day compressive strength of samples. Moreover, it seems that the 28-day strength values of the mortars with colemanite significantly decrease according to standard mortars.
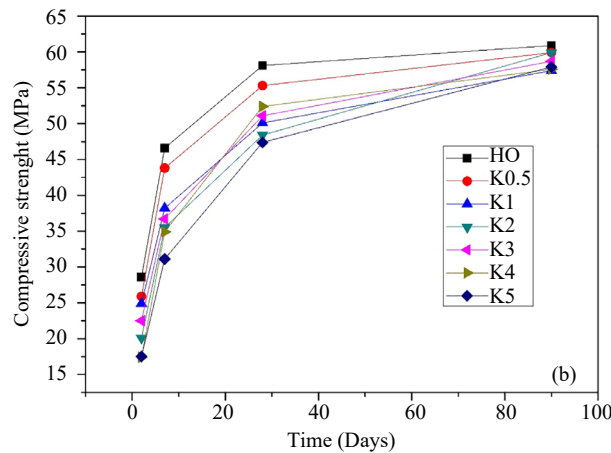
Figure 3. Compressive strength of Cement-A, the cements with added colemanite

In cement, one of the most important mechanical properties sought in cement is high compressive strength. The 2-, 7-, 28- and 90-day compressive strength of Cement-A mortars with 5% colemanite, 5% ulexite and without boron mineral are seen in Figure 4. The ulexite reduced the early-age compressive strength of the cement more than colemanite. At the early age, because of the initial setting time, the problem of the samples with ulexite, the compressive strength values of the samples with added colemanite and ulexite are highly different from each other, but after the 7th
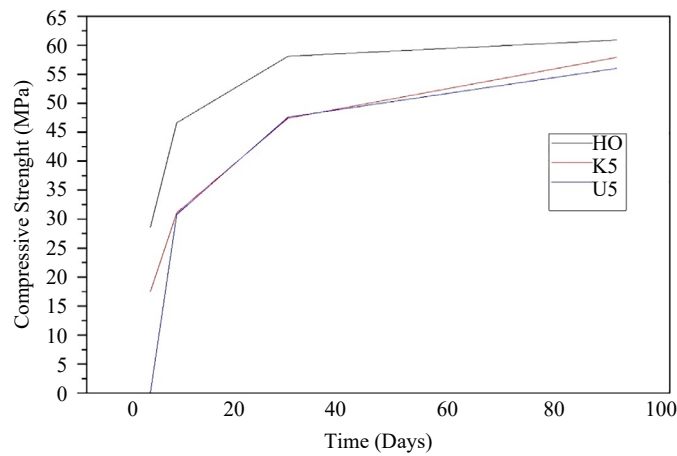
day, their values are very close to each other.

Figure 4. The variation of compressive strength of Cement-A depending age of mortars with 5% colemanite and 5% ulexite and standard (H0) sample

Figure 5 presents the compressive strength ratios obtained by dividing the 2-, 7-, and 28-days compressive strength by 28 age, the compressive strength for Cement-A, Cement-B, and Cement-C. The changes of the initial and final setting times and compressive and flexural strength are similar to each other in Cement-A, Cement-B and Cement-C. There is no difference between the mechanical and physical properties of the cement produced with the colemanite-containing clinker, which was obtained by adding colemanite into farina, and the cement produced by directly adding colemanite. As seen in Figure 5, cement groups that added colemanite show similar behaviours, and as the colemanite addition ratio increases, the compressive strength of cement decreases.
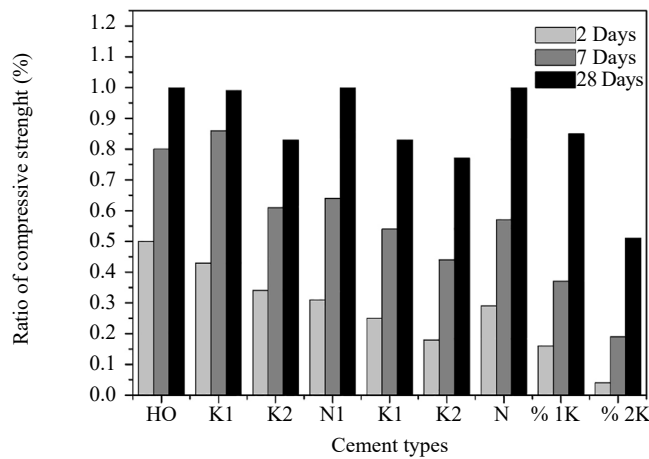
Figure 5. The proportional change of the compressive strength according to the cement types

Figures 6, 7 and 8 present the image obtained from a scanning electron microscope for the cement paste without boron mineral, with 2% added colemanite and with 2% added ulexite, respectively. The results show that different formations are found between the hardened cement paste without additives and the sample with added colemanite and ulexite (Figures 6, 7, and 8). Figure 7 shows that thin needles are formed in the samples with added colemanite.
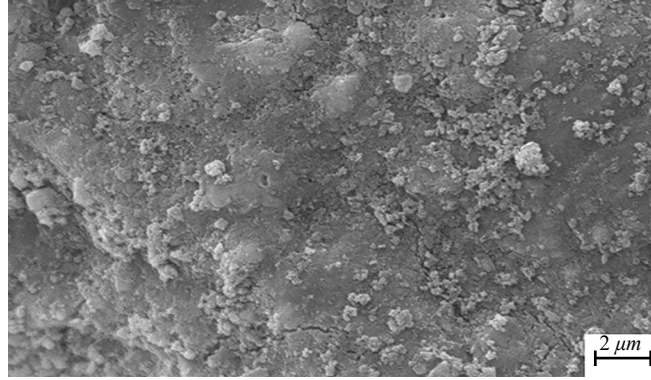
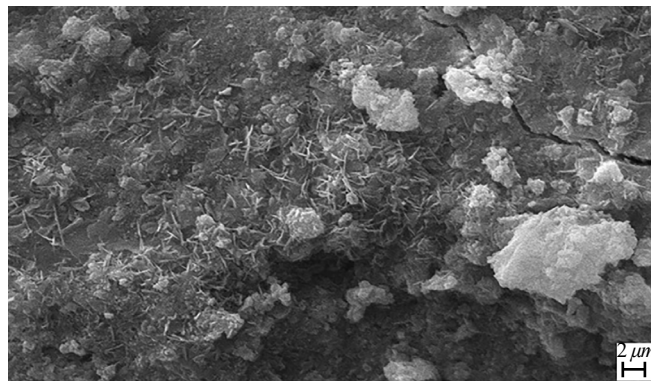**Figure 6.** The SEM image of the hardened cement paste without additive (cement A) (× 5,000)

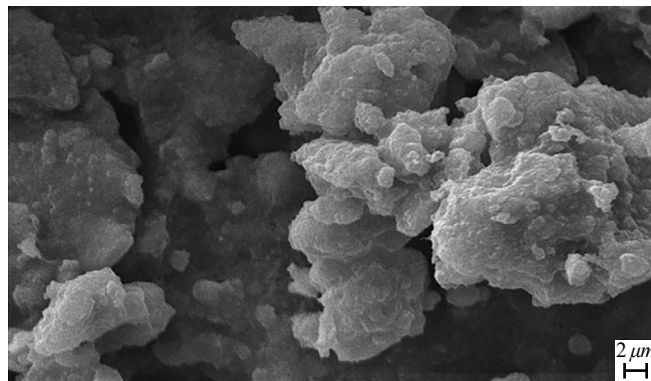**Figure 7.** The SEM image of the hardened cement paste with 2% added colemanite (Cement A) (× 5,000)

Figure 8. The SEM image of hardened cement paste with 2% added ulexite (cement A) (× 5,000)

The micro-structural results obtained by SEM show that different internal structures are formed in the hardened cement paste with colemanite and with ulexite without boron during the cement hydration.

Figures 9 and 10 show the variations of the hydration heat of the cements with added colemanite, calculated with three and seven days hydrate cement, respectively. As the colemanite ratio in the cement increases the hydration heat of the cement decreases by both three and seven days. It is observed that the hydration heat of cement with 3% colemanite decreases as 27% according to the cement without boron minerals for three-day (see Figure 9). Also, the melting heat of the dry cement with 10% colemanite is 5.65% less than the melting heat of the cement without colemanite (see Table 3). Moreover, it was found that the melting heat of the three-day samples was higher than the seven-day samples (see Table

3) because the colemanite decelerates the hydration of the cement.

Table 3. Hydrate heat values of the colemanite-containing cements (J/gram)

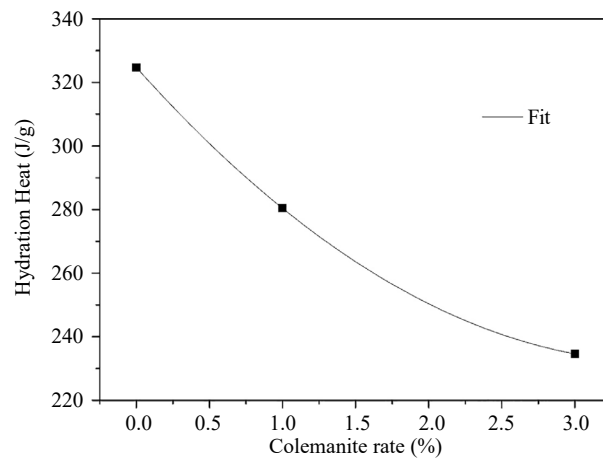| Colemanite addition rate (%)	0	1	3	5	10
Melting heat of dry cement (Q_a)	2,245.0	2,226.8	2,211.2	2,171.6	2,118.1
Melting heat of hydrate cement for 3 days (Q_i)	1,920.3	1,946.4	1,976.6	-	-
Melting heat of hydrate cement for 7 days (Q_i)	1,874.0	1,860.8	1,849.0	1,831.0	-
Hydration heat for 3 days (H_i)	324.7	280.4	234.6	-	-
Hydration heat for 7 days (H_i)	371.0	366.0	362.2	340.6	-

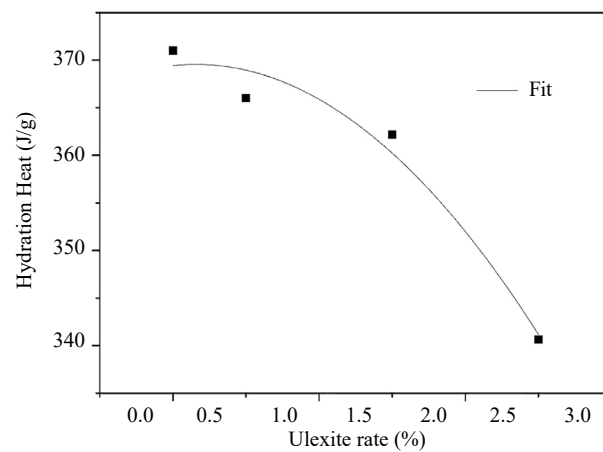**Figure 9.** Hydration heat calculated with the three-day hydrate cement

Figure 10. Hydration heat calculated with seven-day hydrate cement

The hydration heat values of the cements prepared by adding colemanite as 1%, 3%, 5% and 10% into the CEM I 42.5 *R* cement are given in Table 3 for 3-day and 7-day. The melting heat of the dry cement was calculated by Eq. 2 by

using ΔT_c of dry cement. The cement with 5% and 10% colemanite didn't set the melting heat of these samples was not measured for three-day and seven-day (see Table 3).
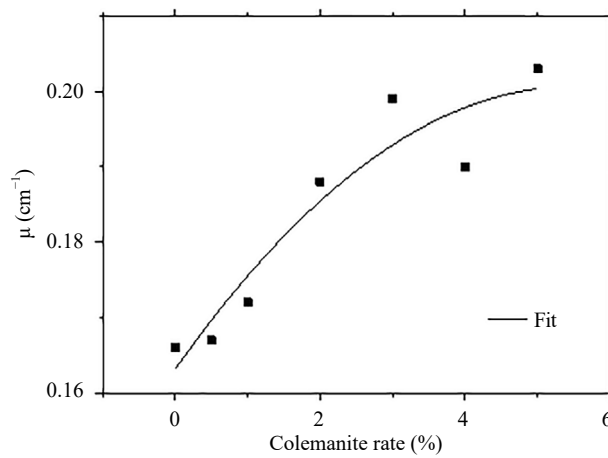

Figure 11. The change of the linear attenuation coefficient versus % additive in the concrete samples with added colemanite for the ^{137}Cs point source (661.6 keV)
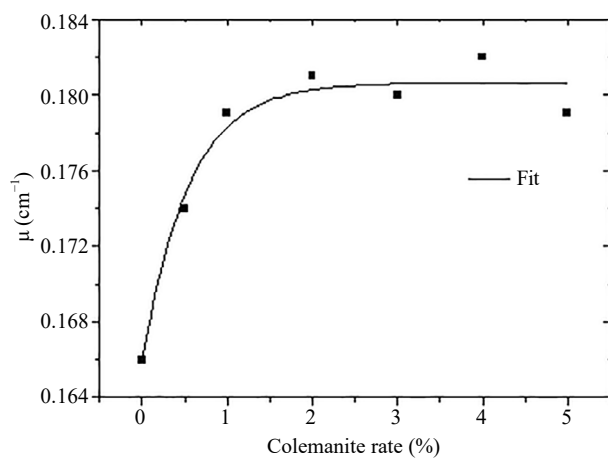

Figure 12. The change of the linear attenuation coefficient versus % additive in the concrete samples with added ulexite for the ^{137}Cs point source (661.6 keV)

Figures 11 and 12 present the variations of the linear attenuation coefficient measured with a Cesium source with the colemanite and ulexite additive ratios, respectively. As the colemanite and ulexite additive ratios increased, the linear attenuation coefficient rose up to when colemanite and ulexite additive ratios were $\leq 2\%$. After this value, the linear attenuation coefficient is almost constant depending on the diminished density of mortars. It is concluded that colemanite and ulexite decreased the radioactive permeability of the mortar.

4. Conclusion

In this study, the following issues were identified.

1-Even a small amount of colemanite and ulexite extended the initial setting time extensively. This indicates that colemanite and ulexite-containing wastes can be used as set retarders.

2-The addition of colemanite caused a significant decrease in the hydration heat of cement at the end of three days.

At the end of 7 days of hydration, there was an approximate proportionality between the hydration heat decreasing ratio and the amount of colemanite used.

3-There is no significant difference in the initial and final setting times and compressive strength regarding the addition of colemanite and ulexite to the cement directly to the clinker during grinding or during the farina baking.

4-Ulexite extends the initial setting time of cement more than colemanite does, for the same ratios used when using the same ratios.

5-A very small amount of ulexite significantly increased the initial setting time of cement and decreased the expansion. Ulexite can be used as a set retarder and expansion-reducing cement or concrete additive.

6-The addition of colemanite and ulexite increased the linear attenuation coefficient of mortar. Thus, colemanite and ulexite decreased the radioactive permeability of mortar significantly.

Conflict of interest

The author declares there is no conflict of interest at any point with reference to research findings.

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