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Contribution to The Manufacture of Petroleum Cement

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DOI : <https://doi.org/10.47485/2766-2624.1046>**Abstract**

This work is devoted to the study of the possibility of valorizing uncalcined barite for the manufacture of petroleum cement, thus minimizing CO₂ emissions resulting from the decomposition of the carbonate during cooking.

We propose to study the effect of adding this ore on the CRS clinker. The samples are prepared by substitution of 10, 20 and 30% barite on the CRS clinker. The mixtures are hydrated and characterized by X-ray fluorescence.

Measuring the setting time of mortars, carried out by the Vicat device, shows that the start and end of setting are proportional to the addition of barite. The compressive strength of the mortars of the different mixtures, measured by the compression device, shows acceptable results with 10% barite.

Keywords: Clinker CRS, Barite, Mortar, Workability and Resistance.**Introduction**

Cement is an essential material for construction, widely used across the world. The manufacture of cements was developed by the arrival of new minerals, either natural, or in the form of waste (cement additions) such as pozzolan, silica fumes, fly ash and blast furnace slag (Chabi et al., 2013). These mineral additions have hydraulic properties (formation of stable hydrated compounds by reaction with water) and pozzolanic properties (formation of stable hydrated compounds that are very poorly soluble in water at ordinary temperature by combination with lime and in the presence of water) interesting (Harichane et al., 2011; Ghrici et al., 2007; Ezziane et al., 2007). It is a fine powder composed mainly of limestone, clay and other minerals. Cement production involves the extraction, processing and mixing of raw materials, followed by high temperature calcination of the mixture to produce clinker (Andrew 2017). The use of recovered and recycled industrial residues, such as cement additions and natural resource deposits in the production of Portland cements have largely solved the problem of national self-sufficiency, as well as that of lower energy costs (Kaid et al., 2010). Algeria is an oil producing country, the oil production operation necessarily involves a phase of cementing oil wells to consolidate them. This cementing phase consumes enormous quantities of specific cement which are usually called class G and H petroleum cements (Al-Yami et al., 2015). Petroleum cements must meet specific criteria according to the American Petroleum Institute

(API) to meet technical requirements. Portland cement types G and H are commonly used worldwide for oil and gas well cementing operations (Arjomand et al., 2018; Bediako et al., 2015; Broni-Bediako et al., 2015). In Algeria, class G portland cement is the only one used for hydrocarbon exploitation (Nelson et al., 2006).

The construction of a well is carried out according to a well-established program, the first step of which is drilling. Then a steel tube designated by the Anglo-Saxon term (Casing) is lowered into the drilled area. This casing exists in several diameters ranging from 30 inches to 4 ½ inches, its function is to support and cover the wall of the well. Cement is then used during these drilling operations to fill the annular space, which is on the order of a few centimeters, between the casing and the surrounding rock formation.

The roles of the cementing material used for the construction of a well are multiple. In particular, this material must:

- Prevent the hole from collapsing and protect the casing from being crushed by the ground
- Anchor the various casings on which the safety equipment is fixed at the wellhead
- Reduce corrosion of casings by fluids from geological formations.
- To isolate the geological formations crossed by an oil well, in a permanent and lasting manner.

Cementing is, therefore, a very important and judicious operation: Its success is a determining factor for the continuation of the next phase; it conditions the water tightness and mechanical integrity of the well, which must be able to withstand the course of its existence. Since time and until today, several authors have developed methods to study the long-term mechanical durability of the cement sheath (Backe et al., 1999; Bosma et al., 1999; Ravi et al., 2002).

The behavior of Portland cement (hydration kinetics, compressive strength) under downhole conditions (temperature and pressure) is very different from that observed under usual conditions. In recent years, new challenges have emerged during the exploitation of increasingly critical deposits, where conventional cement formulations present limitations in the face of many constraints including stability during pumping, mechanical strength, degradation. of cement to acidic and carbonate environments and the drop in compressive strength in high salinity environments (Senhadji et al., 2014; Khalifeh et al., 2017).

This experimental work studies the possibility of partial substitution of CRS clinker with barite ore to produce petroleum cement, at various percentages (0%, 10%, 20% and 30%).

In this study, we study the effect of barite on the mechanical behavior of the synthesized cement in order to arrive at a relative contribution to the manufacture of petroleum cement

by comparing these properties obtained by the properties required by API standards (American Petroleum Institute) which are interested in the specifications of cements used only for wells. This allows us to contribute to the valorization of local materials in the cement industry in order to obtain a new petroleum cement.

Materials and Methods

Preparation, analysis and characterization of the materials used

Clinker Portland CRS

Product obtained by mixing natural raw materials (approximately 70% limestone, 30% clay and 1% iron ore) produced by the GICA group of Ain-Touta located in the wilaya of Batna (Algeria) and calcined (clinkerization) at 1450°C. It appears in the form of hard, crystallized nodules and is made up of anhydrous calcium silicates and aluminates, resistant to sulfates whose C3A = 4.68.

Gypsum

Obtained from SCIMAT of AIN Touta. It is a common saline rock of sedimentary basins prone to subsidence, it is mainly composed of the ore gypsum, a very common saline ore of the sedimentary series, and doubly hydrated calcium sulfate. The latter is both a chemical species and a mineral species, described by the formula $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The percentage of gypsum used in the manufacture of cement does not exceed 6%.

Elements %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	CL
Gypsum	8,50	2,54	1,04	29,32	3,07	36,53	0,53	0,03	0,008

Table 1: Chemical composition of gypsum.

Sand

Standardized sand is packaged in polyethylene bags, each containing 1350 g ± 5g.

The particle size composition of this sand, determined by sieving, is:

CEN certified conforming to EN 196-1.

ISO compliant with ISO 679: 2009.

Physical parameters	Values
Absolute density	2.64g/cm ²
Sand equivalent	92%
Finesse	2.55
Absorption coefficient	0.5
Silica content	98.06
Nature	Natural rolled

Table 2: Physical characteristics of sand.

Mixing water

It complies with standards NF P 18-325 and NF P 18-303.

Ca ²⁺	Mg ²⁺	K ⁺	Na ⁺	Cl	NO ₃ ⁻	SO ₄ ⁻	HCO ₃ ⁻	Ph
104.21	88.95	3.26	49.25	134.9	5.14	205.9	323.3	7.18

Table 3: Chemical composition of water.

Barite

Barite is an ore composed of barium sulfate (BaSO_4), from ENOF (National Company of Non-Ferrous Products) which exploits the Ichemoul deposit (Batna City) and refining is done at the Ain-Unit. Mimoun (Khenchela City). It is commonly found in various sedimentary rocks and is often found as veins or nodules in limestone and dolomite. Barite has a high specific gravity, making it valuable in various industrial applications.

	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	Cl	CO_2
Barite	2,67	0,09	0,07	0,05	7,09	0,11	0	14,78	0	1,11

Table 4: Chemical composition of barite.

Preparation, making and conservation of study mortars

The mortars were made in a standardized mixer with a capacity of five liters, the mixing sequence used is that prescribed by standard EN 196-1 (NF EN 196-1, 1995). The operations for placing the test pieces were carried out in accordance with the operating procedure described by the standard by the quality laboratory of the GICA cement plant (Algeria). In order to monitor the mechanical behavior and durability of the mortars, we considered different compositions illustrated in Table 5.

Samples	Clinker %	Gypsum %	Barite
CRS A	95	5	/
Sample B	85	5	10
Sample C	75	5	20
Sample D	65	5	30

Table 5: Percentage composition of cement samples.

- Barite dosage (0%, 10%, 20% and 30%)
- Ratios C/S=1/3, E/C=0.5.

In the anhydrous state, the cements underwent chemical treatments to determine the density as well as Blaine SSB density and specific surface area tests according to standards EN 196, NF EN 196-1 and EN 196-6 respectively.

The physical properties studied are: the normal consistency and the start and end time of setting determined by the Vicat test of pure pastes of compound cements used in accordance with standard NF 196-3.

The mechanical properties studied of the prepared mortars are the resistance to compression and bending on standardized test pieces $4 \times 4 \times 16 \text{ cm}^3$ according to standards NF P-18-406 and NF P18-407.

After unmolding at 24 hours of age, the test pieces are stored in a humid environment (20°C and 100% RH) until the age of the test at 7 and 28 days according to standard NF P15-402.

Results and Discussions

Influence of normal consistency on synthesized cements

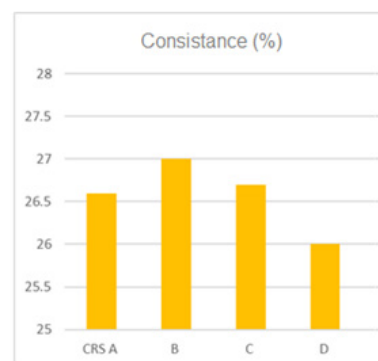


Figure 1: Normal consistency test results.

The demand for water appears to be increased when there is a high content of carbon contained in the cement given that carbon is also porous but finer than silica. The water demand also increases with grinding time to reach a maximum and then decreases during pronounced grinding. This increase in water demand can compensate for the decrease in workability.

Influence of the barite substitution rate on the setting of synthesized cements

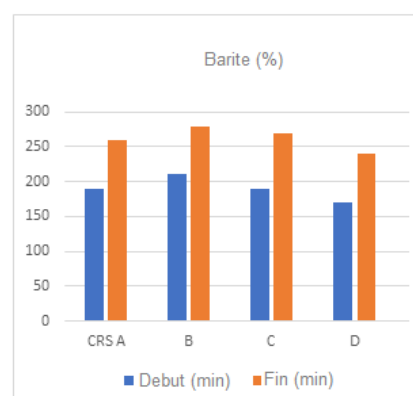


Figure 2: Results of the start and end of setting.

Figure 2 shows that the synthesized cements have a time close to that of Portland CRS cement.

In fact, we ground the CRS clinker with 5% gypsum. The setting time evolves with a slight slope as the percentage of barite increases.

This setting time is due to the addition of gypsum to the synthesized cement mixture. This delay can be explained by the existence of sulfates in the barite which reduces the rate of hydration of the alite (C3S). The latter forms an obstacle to the progression of hydration (K. Murakami 1969) This is explained by the slowness of the pozzolanic reaction (Ghrici et al., 2007).

Influence of density on the paste of synthesized cements

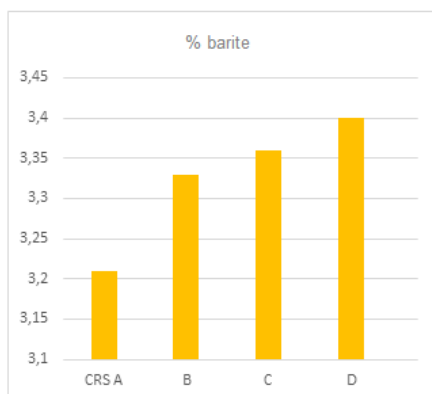


Figure 3: Effect of Barite addition rate on density.

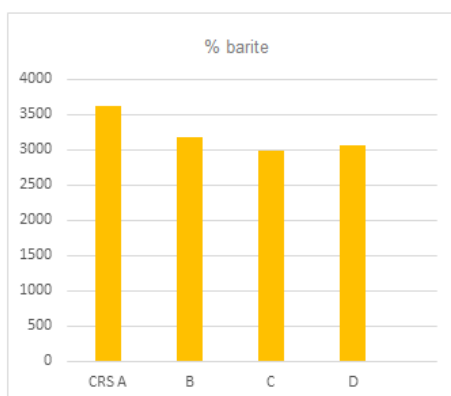


Figure 4: Effect of Barite addition rate on Blaine specific surface area.

It is noted that the Blaine specific surface area of the synthesized cements decreases as a function of the increasing rate of substitution of the CRS clinker up to 20% substitution, and rises a little from 30% substitution, this may be due to the nature of the incorporated substitution (Barite).

The density of synthesized cements is greater than that of Portland CRS cement. As a result, if a part of CRS cement is replaced with barite in relation to the mass, the paste volume of the mixture decreases.

Influence of loss on ignition on the paste of synthesized cements

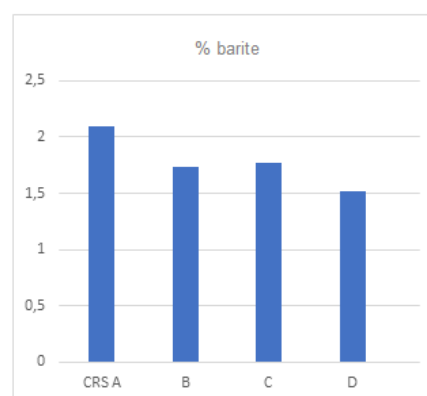


Figure 5: Loss on ignition of synthesized cements.

According to API standards, the maximum allowable percentage of loss due to ignition is 3%. Samples B, C and D had a random loss on ignition, lower and close to the reference sample A, which is 2.11%.

Mechanical behavior of mortars

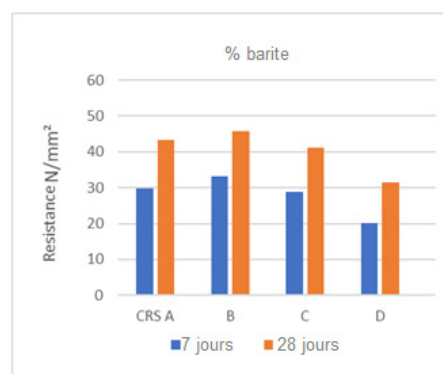


Figure 6: Compression test results.

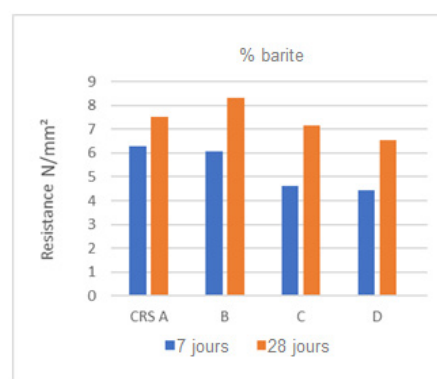


Figure 7: Bending test results

Both Figures 6 and 7 provide certainty that the resistance of mortars evolves by increasing over time and with a decreasing rate of substitution. These developments are clearly visible on the graphs of the resistances in bending and compression. We can see that the control mortar, the first mixture with 100% CRS

cement, has for each of the ages a resistance greater than those of the other mortars of 20 and 30% substitution except that with a substitution of 10% barite which has better resistance to compression and bending. But on the other hand, at the age of 28 days, the control mortar has the best bending resistance.

In fact, the evolution of the resistances depends on the barite content and the storage time of the specimens, as well as the C3S. Therefore, barite could have an effect on the reactivity of the CRS clinker in the short term, manifested by an increase in setting time and a reduction in resistance (G.K. Moir 1982).

Conclusion

The general objective of this work was to evaluate the effect of substituting CRS cement with barite powder to have a new petroleum cement on mechanical strength with that of class G using ratios $C/S=1/3$ and $E/C=0.5$. The advantage of the approach is to allow the substitution of barite without calcination, thus reducing greenhouse gas emissions. Substituting 10% of CRS cement with barite has been reported to significantly reduce CO_2 emissions and provide better mechanical properties.

The first results presented here reveal that the taking times of all samples lie on a slightly convex curve. The use of barite usually reduces water demand. The consistency decreases with the barite content, suggesting an excess of water which can be explained by the difference in specific surface area between the cements synthesized by barite and the CRS cement and by the lower absorption of the mixing water by the barite, which reduces the quantity of free water in the cement matrix.

Barite powder modifies the kinetics of development of the compressive strength of mortars. It leads with a rate of 10% substitution and a slow development of resistance at the age of 28 days which is better than that of the control sample without barite. This observation can be explained by the modifications in the hydration of the matrix induced by the substitution of cement by barite which absorbs less water than cement.

The resistance to bending with a rate of 10% substitution is improved at 28 days as well as at 7 days of setting. Barite powder therefore exerts a physical and chemical effect (pozzolanic and alkali-silica reaction).

The use of barite powder as a partial substitute for CRS cement in commonly used mortars seems feasible. However, additional work remains necessary to confirm these initial results.

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