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Experimental Study of the Effect of Clinker Substitution Rate by Dolomite on the Physico-Mechanical Properties of Portland Cement

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Abstract: Reducing the carbon footprint of cement has generated a significant amount of research in the cement industry. Substituting a portion of the clinker with mineral additives remains one of the least expensive and easiest solutions to implement in the cement industry. One of the least used mineral additives is dolomite, which is a natural and abundant resource. However, the rate of substitution of clinker by dolomite can alter the physico-mechanical properties of cement. The objective of this study is to experimentally determine the optimal cement formulation using dolomite as a mineral additive. To this end, a sample of cement without additives (CPA) and five samples of cement with dolomite added at different rates (20%, 25%, 30%, 35%, and 40%) were developed from co-grinding clinker, dolomite, and gypsum. A chemical analysis by XRF of the raw materials used was carried out and the evolution of the physico-mechanical properties of the cement as a function of the dolomite content in the different samples in anhydrous and hydrated state was monitored from different tests. The composition with a 25% clinker substitution rate was retained as the optimal formulation for a Portland cement with dolomite.

1. Introduction

In Côte d'Ivoire, various investments in infrastructure and housing construction programs in recent years have led to increased demand for cement. In grinding units, Portland cement is manufactured by grinding a mixture containing 95% clinker and 5% gypsum (Logbi, 2019), (Zeraoui, 2020). Clinker is a basic component in cement manufacturing (Bisulandu and Marias, 2019) because it provides cement with its main characteristic, hydraulicity, i.e., its ability to harden when it reacts with water (Chen *et al.*, 2010; Jabri *et al.*, 2012; Yahyaoui *et al.*, 2014; John and Lothenbach, 2023). However, clinker production has two major drawbacks: it is a source of pollution (Gauffinet, 2016) and consumes enormous amounts of energy (Fergani, 2016). The partial or total replacement of clinker with substitute materials in cement manufacturing has greatly contributed to reducing its carbon footprint by up to 50% (Dumé, 2022) and has led to enormous energy savings. The use of substitute materials for clinker therefore has a positive impact on the cost of cement manufacturing and environmental protection (Deboucha *et al.*, 2012). Furthermore, replacing clinker with these materials must not affect the physical and mechanical properties of Portland cement. Some studies have shown that dolomite powder, a natural material abundant in the Earth's crust, can be used as a filler in concrete manufacturing as a partial replacement for cement. The objective of this work is to experimentally determine the optimum rate of clinker substitution by dolomite in cement manufacturing. This optimization was carried out using various physical and mechanical analyses performed on different samples of cement in its anhydrous state (absolute density, Blaine specific surface area), in its hydrated state (standard consistency, setting time, and stability test) and in the form of standard mortars (mechanical compressive strength), while varying the substitution rate of clinker by dolomite and maintaining the gypsum content at 5%.

2. Materials and methods

The various cement samples were prepared and analyzed at the quality control laboratory of the LAFARGE HOLCIM ABIDJAN cement plant in Cote d'Ivoire.

2.1 Analysis of raw materials

The raw materials (**Figure 1**) were dried at 100°C for 24 hours before being used. They underwent chemical analysis by X-ray fluorescence, and their apparent densities were determined in accordance with standard NF EN 196-6.

Clinke **Gypsum** **Dolomite**

Figure 1. Raw Materials

2.2 Cement production

These raw materials were ground using a ball mill in order to replicate industrial cement production conditions. Six samples of different formulations were produced: a control formulation of CPA cement (95% clinker + 5% gypsum) and five formulations in which the clinker was replaced by dolomite at different rates (20%, 25%, 30%, 35%, and 40%) while maintaining the gypsum content at 5%. These samples were designated by the codes D20, D25, D30, D35, and D40, respectively (**Figure 2**).

CPA **D20** **D25** **D30** **D35** **D40**

Figure 2. The cement samples prepared

2.3 Physical characterization of cement samples

The cement samples were subjected to various physical analyses.

- **Absolute density**

The absolute density ρ_ab of the cement samples was measured using a pycnometer, also known as a Le Chatelier densimeter, in accordance with standard NF EN 196-6.

- **Specific surface area or Blaine surface area**

The specific surface area or Blaine surface area of the samples was measured in accordance with standard EN 196-6 using a Blaine permeameter.

- **Standard consistency, setting time, and expansion**

The standard consistency, setting time, and expansion of the cement pastes from the different cement samples were determined in accordance with standard EN 196-3.

- **Mechanical tests: measurement of mechanical strength**

Compressive strength tests were carried out in accordance with standard NF EN 196-1.

3. Results and Discussion

3.1 Analysis of raw materials

3.1.1 Apparent densities

The apparent density of the raw materials used is given in **Table 1**. Clinker has the highest bulk density. This means that clinker has a higher grain density than dolomite and gypsum, resulting in greater compactness in pure clinker-based cement formulations. Clinker is a highly reactive material and is the main component of cement. After hydration, tricalcium silicate (C_3S) and dicalcium silicate (C_2S) form hydrated monocalcium silicate (C-S-H) crystals, which, by bonding with each other and with the constituents of the material in which they were formed, give the material its strength (Abdo, 2009). Dolomite has a slightly lower density than clinker. This corroborates its use in cement as an inert mineral additive and filler. Its role in cement formulation is therefore to reduce the proportion of clinker, thereby positively impacting the environment by lowering the carbon footprint. The presence of dolomite can also have a positive impact on the rheological properties of concrete (Mosser, 2023). The density of gypsum is similar to that of dolomite. The proportion of gypsum in cement is low. Its role is to prevent the cement from setting too quickly (Souchu, 2010).

Table 1. Density of Raw Materials

Raw Materials	Clinker	Dolomite	Gypsum
Apparent densities (g/L)	1594	1511	1504

3.1.2 Chemical analysis of raw materials by X-ray fluorescence (XRF)

The chemical composition of the raw materials and the mineralogical composition of the clinker are given in **Tables 2** and **3**, respectively. Tricalcium silicate (C_3S) and dicalcium silicate (C_2S) account for approximately 75% (more than 2/3) of the mass of the clinker used. Its mass ratio (CaO)/(SiO_2) is equal to 3.12 (<2) and its MgO content is equal to 1.47 (<5%) (Bouchenafa, 2019). The CaO (27.92%)

and MgO (26.48%) content is typical of dolomite with the formula $CaMg(CO_3)_2$. There is a certain amount of impurities. The gypsum used. with the formula ($CaSO_4 . 2H_2O$). has a suitable CaO (28.93%) and SO_3 (35.40%) content. The chemical composition of the gypsum used therefore indicates that it is of natural origin. Chemical analysis by X-ray fluorescence has shown that the chemical and mineralogical compositions of the clinker. dolomite. and gypsum used to prepare the various cement samples meet the quality criteria for raw materials used in the cement industry.

Table 2. Chemical composition of raw materials

Matières premières	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	PAF	SO_3	Na_2O	TiO_2	P_2O_5	ZnO
Clinker	64.6	20.70	5.21	4.12	1.47	0.42	2.48	0.34	0.22	0.28	0.05	0.05
Gypse	28.93	3.67	1.21	0.472	1.00	0.546	35.4	29.72	0.041	0.056	0.016	0.03
Dolomite	27.92	10.92	1.03	0.689	26.83	0.545	31.47	0.168	0.017	0.047	0.066	0.02

Table 3. Mineralogical composition of clinker

Mineral phase	Percentage
C_3S	64.088
C_2S	10.995
C_3A	6.828
C_4AF	12.539

3.2 Physical properties of the cement samples produced

3.2.1 Absolute densities of cement samples

The absolute density of the different cement samples is given in **Table 4**. The results recorded in the table were used to plot the variation curve of absolute density (**Figure 3**) versus the dolomite content in the cement sample. According to Guiraud, (2018), the absolute density varies from 2.90 to 3.15 g/cm^3, depending on the type of cement. The absolute densities of the different cement samples used are therefore in accordance with the standard.

Table 4. Absolute density of samples

Echantillon	Masse volumique absolue (g/cm^3)
CPA	3.06
D20	2.94
D25	2.94
D30	2.93
D35	2.93
D40	2.93

CPA Portland cement has the highest density value (3.06 g/cm^3). This value is to be expected, as this material is 95% clinker, which is denser than dolomite and gypsum. For the other samples, in which part of the clinker has been replaced by dolomite, the absolute density of the cement generally decreases as the dolomite content increases. However, a plateau is observed, reflecting the relative stability of these values, which vary between 2.94 and 2.93 g/cm^3. The small difference between these variations in density co uld be explained by the fact that the clinker is replaced with the same material.

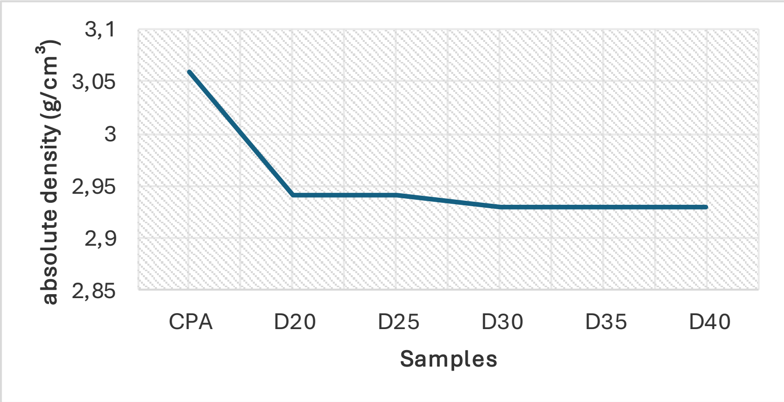

Figure 3. Curve showing the variation in absolute density as a function of the dolomite Curve of variation of the specific surface area of cement samples

3.2.2 Specific surface area

The specific surface area values of the cement samples are recorded in **Table 5**.

Table 5. Specific surface areas of different cement samples

Samples	Specific area (cm^2/g)
CPA	4530
D20	5412
D25	5694
D30	6325
D35	6357
D40	6198

These specific surface area values were used to plot the variation curve of the specific surface area (**Figure 4**) as a function of the dolomite content in the different cement samples.

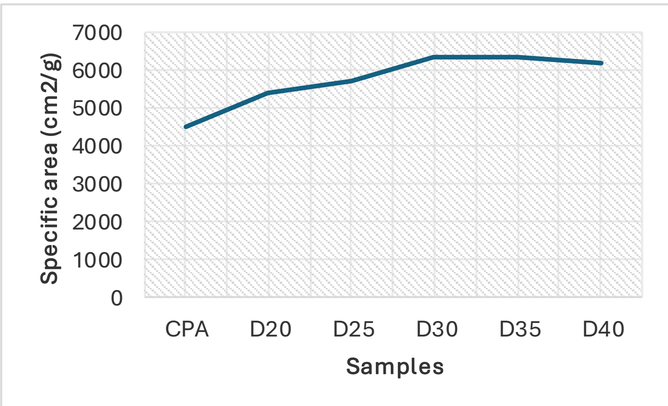

Figure 4. Curve of variation of the specific surface area of cement samples.

The CPA cement sample has the lowest specific surface area compared to the other samples. This can be explained by the absence of finer mineral additives such as dolomite in this sample. In general, the specific surface area of the cements produced, and therefore their fineness, increases with the amount of dolomite added to the samples, up to 35%. This promotes rapid hydration of these samples, causing the cement to set quickly (Mounanga, 2004). Sample D35 has the highest specific surface area, reflecting increased particle fineness. This characteristic implies greater reactivity, as a large specific surface area promotes chemical exchange with water, thereby accelerating hydration reactions and improving the mechanical properties of the cement. Beyond 35% dolomite addition, the specific surface area of the cement samples decreases. There is, therefore, a threshold that should not be exceeded when substituting clinker with dolomite in cement formulation.

3.2.3 Standardized consistency, setting time, and expansion

The standardized consistency defined by the water/cement ratio (W/C), setting time, and expansion of the different cement samples are recorded in **Table 6**.

Table 6. Physicochemical characteristics of cement pastes

Samples	W/C (%)	Initial setting time (min)	Setting time (min)	Expansion (mm)
CPA	25.6	172	217	0.5
D20	26.8	153	198	0.5
D25	28.46	167	217	1
D30	27.8	201	251	1
D35	28.4	200	250	1
D40	27.2	204	254	1.5

The CPA control sample has a W/C ratio of 25.6%. while that of samples D20 and D25 increases to 28.5%. This reflects a higher water demand for these samples in order to maintain the same standardized consistency. Between 30 and 40%. the W/C ratio remains high. It can be deduced that. in general. the presence of dolomite in cement increases the water demand to maintain the same workability.

The setting of the CPA sample begins at 172 min and ends after 217 min. For samples D20 and D25. setting begins more quickly (153 min and 167 min. respectively) compared to CPA and also ends more quickly (198 min and 217 min. respectively). This acceleration in cement setting is due to the filler effect. which results from the influence of the hydration kinetics of the major phases of cement in the presence of mineral additives such as dolomite (Mejdi, 2019). From a 30% substitution rate of clinker by dolomite (samples D30, D35 and D40). longer setting and finishing times are observed compared to CPA. This is due to the dilution effect of the active clinker.

The expansion of the different cement samples increases with the substitution rate but remains low overall compared to the EN 196-3 standard (<10 mm). A slight increase is observed for sample D40. which is due to a high free magnesia content in this sample (MgO).

3.2.4 Mechanical resistance to compression

The measured compressive strength values of the different cement samples, expressed in MPa, are given in **Table 7**. These values were used to plot the comparative graph in **Figure 5**. After two days, the various cement samples with added dolomite generally exhibit acceptable early-age mechanical strength values compared to the CPA. This is due to the filler effect of the added dolomite, which improves the compactness of the mixture and has a favorable effect on cement hydration and therefore on early mechanical strength. After 7 days, a rapid increase in mechanical strength was observed for all six samples, indicating good cement hydration for each of the formulations. Samples D20, D25, and D35 showed mechanical strength values higher than those of the CPA. This suggests that the additions were made in the correct proportions.

Table 7. Mechanical strength of cement samples

Samples	2 days	7 days	28 days
CPA	25.3	39.0	50.8
D20	27.7	41.4	50.8
D25	29.8	42.8	50.2
D30	23.5	36.7	44.6
D35	26.4	39.6	37.3
D40	26.1	27.3	44.8

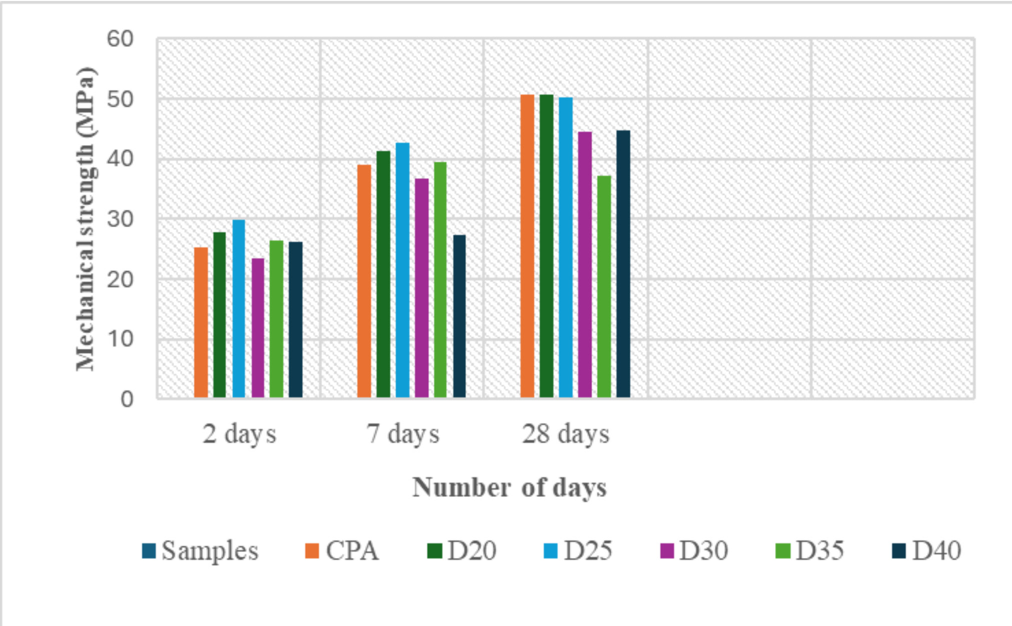

Figure 5. Comparative graph of mechanical strength at different ages

At 28 days, CPA cement reaches a mechanical strength value of 50.8 MPa. This value complies with high mechanical strength classes, more specifically strength class 52.5 (Guiraud, 2005). Sample D20 achieves the same mechanical strength value (50.8 MPa) as CPA, and sample D25 achieves a strength value of 50.2 MPa. These two cement samples achieve virtually the same mechanical strength value as CPA. These samples (D20 and D25) perform well mechanically despite the reduced clinker content compared to CPA. The best compromise between reactivity and dolomite addition is achieved for sample D25, which achieves the highest mechanical strength values at all ages. A decrease in mechanical strength is observed for samples D30, D35, and D40, which have strength values of 44.6 MPa, 45.5 MPa, and 44.8 MPa, respectively, significantly lower than that of the CPA. Replacing between 30% and 40% of the clinker results in a loss of mechanical strength. This may be the result of a dilution effect of the fine particles (Mosser, 2023).

Conclusion

The optimal formulation of cement using dolomite as a mineral additive was determined based on various physicochemical tests on cement samples, in anhydrous or hydrated form, containing different percentages of dolomite (0%, 20%, 25%, 30%, 35%, and 40%). The results showed that the rate of clinker substitution by dolomite has an impact on the physicochemical properties of cement, which are altered above a dolomite content of 25%. Dolomite can therefore be used as an inert mineral additive to reduce clinker consumption in Portland cement manufacturing. However, to preserve the physical and mechanical properties of the cement, clinker substitution with dolomite must be limited to 25%.

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