Influence of Limestone Fillers on Rheological and Mechanical Performance of Concrete

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1. INTRODUCTION

The crushing aggregates industry generates large quantities of more or less fine waste [1-3]. These fines are often a problem for the environment. They indeed present, in the case of a layer in the form of dust which, by their fineness, is easily transported by the air current and leads to physical and aesthetic pollution of the environment.

For some time, researchers have been actively concerned about the possibilities of using limestone crushing aggregates, despite the mistrust in some companies surrounding the use of these aggregates in concrete [4, 5]. Much of this distrust is linked to the presence of large quantities of fillers and the action of these fillers on the properties of fresh and hardened concrete.

By their fineness and by their more or less significant reactivity in the presence of cement, mineral additions generate significant modifications on the properties of cementitious materials in the fresh and hardened state [6-8]. In the fresh state, the presence of mineral additions modifies the structure of the granular skeleton and the friction between the solid components in the liquid phase. During setting and hardening, the particles of the additions interact in the cement hydration process by modifying the structure of the hydrated products and for some can react chemically in cement medium to form new hydrated products which have an additional binding character. [9, 10]. The mechanisms at the origin of these modifications seem particularly complex, however several recent studies [11-14], agree to distinguish the main effects of additions in a cementitious material.

Industrial co-products such as fly ash, silica fumes, blast furnace slags, have been of a positive contribution thanks to their hydraulic and pozzolanic characteristics [15-28]. Their use has been generalized as an additive in durable concretes and high-performance concretes [29-44]. However, the price of these additives is relatively high, which is not the case for limestone and siliceous fillers.

The tolerated limits for filler content in concrete vary from one country to another, as shown in Table 1.

Table 1. Specifications of fillers from crushing products in different countries (% by mass of sand)

<table>
<thead>
<tr>
<th>Country</th>
<th>Specification</th>
<th>Maximum Allowance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Germany DIN 44 226 1971</td>
<td>&lt;4%</td>
<td></td>
</tr>
<tr>
<td>Canada CSA A231 1973</td>
<td>&lt;3.5%</td>
<td></td>
</tr>
<tr>
<td>Italy Uni 7 163</td>
<td>&lt;3.5%</td>
<td></td>
</tr>
<tr>
<td>Belgium NBN 589-102 1969</td>
<td>&lt;3-5%</td>
<td></td>
</tr>
<tr>
<td>U.S.A ASTM C33 1986</td>
<td>&lt;3-7%</td>
<td></td>
</tr>
<tr>
<td>France NF P 18 541.18 301 NF XP18 540. 1994-1983-1997</td>
<td>12 à 18%</td>
<td></td>
</tr>
<tr>
<td>Algeria DTR BE 2-1 RETAB 1991-1998</td>
<td>4 à 10%</td>
<td></td>
</tr>
<tr>
<td>Great Britain BS 822 1992</td>
<td>&lt;16%</td>
<td></td>
</tr>
</tbody>
</table>

In this experimental study, we are interested in highlighting the possible beneficial effects of limestone fillers from two quarries in the Batna region, namely; the National Aggregates Company (ENG) and the Ain-Toua Cement Company (SCIMAT) on the workability and resistance of the concrete material.

The main objective of this study is to identify the maximum proportions that can be introduced without compromising quality, by optimizing the granular mixture and to highlight possible beneficial effects.

We will study as influential factors the origin of the aggregate, the granular skeleton and the proportion of fines and as properties the density, the content of entrained air, the consistency of the fresh concrete, the mechanical resistances and the dimensional variations of the hardened concrete.

2. EXPERIMENTAL PROGRAM

2.1 Principle of the study

It consists in formulating various common concretes using...
limestone aggregates from a classic base composition using sand de-filtered by washing and sieving at 80μ and introducing fillers in different proportions from 6 to 24% per not 6%.

2.2 Basic materials

2.2.1 Cement

The cement used is of the CEM I 42.5 N type without any addition (95% clinker with 5% gypsum), produced by the GICA group of Ain-Touata located in the wilaya of Batna (Algeria) and conforms to the NF standard. EN 197-1, 2000) whose clinker is produced and ground together with gypsum by the GICA group. The mineralogical and potential chemical (Bogue) compositions of the clinkers communicated by the producer are given in Table 2.

The dosage of natural gypsum (dehydrated calcium sulphate, CaSO₄. 2H₂O) was kept constant at 5%. Its chemical composition is given in Table 3.

2.2.2 Mixing water

It complies with the standards NF P 18-325 and NF P 18-303. The results of the chemical analysis of the water are given in Table 4.

Table 2. Clinker composition %

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Apparent Density (g/cm³)</th>
<th>Absolute Density (g/cm³)</th>
<th>Mold thin (cm²/g)</th>
<th>Time Taken: Start (Hours)</th>
<th>Time Taken: End (Hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.100</td>
<td>3.190</td>
<td>4200</td>
<td>2h: 12</td>
<td>3h: 08</td>
</tr>
<tr>
<td>Mineralogical Composition (Bogue Method)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C₂S</td>
<td>57.83</td>
<td>C₂S</td>
<td>C₃A</td>
<td>16.75</td>
<td>8.03</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical Composition (Fluorescence X)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>19.82</td>
<td>Al₂O₃</td>
<td>Fe₂O₃</td>
<td>3.07</td>
<td>1.16</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sand equivalent (ESV)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7J</td>
<td>47.05</td>
<td>50.30</td>
<td>5.9</td>
<td>7.7</td>
<td>8.85</td>
</tr>
<tr>
<td>2J</td>
<td>47.05</td>
<td>50.30</td>
<td>5.9</td>
<td>7.7</td>
<td>8.85</td>
</tr>
</tbody>
</table>

Table 3. Chemical composition of gypsum %

<table>
<thead>
<tr>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>SO₃</th>
<th>K₂O</th>
<th>Na₂O</th>
<th>CL</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.50</td>
<td>2.54</td>
<td>1.04</td>
<td>29.32</td>
<td>3.07</td>
<td>36.53</td>
<td>0.53</td>
<td>0.03</td>
<td>0.008</td>
</tr>
</tbody>
</table>

Table 4. Phys-chemical composition of mixing water (mg/l)

<table>
<thead>
<tr>
<th>Ca</th>
<th>Mg</th>
<th>O₂</th>
<th>CO₂-H</th>
<th>Cl</th>
<th>SO₄</th>
<th>NO₃</th>
<th>PH</th>
<th>Toughness</th>
<th>T°p</th>
<th>Conductivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.21</td>
<td>44.72</td>
<td>2.5</td>
<td>285.86</td>
<td>97.71</td>
<td>122.84</td>
<td>8.24</td>
<td>7.46</td>
<td>42.64°f</td>
<td>19.3°C</td>
<td>1036μS/cm</td>
</tr>
</tbody>
</table>

2.2.3 Granulate

The granulates come from the Society of SCIMAT Cements of Ain-Touata (Group of Aggregates and Broken Sand). These are dolomitized, crushed and screened limestone materials, marketed as granular classes 4/8, 8/16 and 16/25 (XP Standard P 18-545 Article 10-EN 12620 and EN).

Table 5. Granulate physical, morphological, and mechanical properties

<table>
<thead>
<tr>
<th>Guis</th>
<th>Sand-ENG</th>
<th>Sand-SCIMAT</th>
<th>Gravel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Granular classes</td>
<td>0/4</td>
<td>0/4</td>
<td>4/8</td>
</tr>
<tr>
<td>Physical properties</td>
<td>Density (kg/m³)</td>
<td>2.65</td>
<td>2.60</td>
</tr>
<tr>
<td></td>
<td>Absolute</td>
<td>1.56</td>
<td>1.60</td>
</tr>
<tr>
<td></td>
<td>Apparent</td>
<td>2.6</td>
<td>2.49</td>
</tr>
<tr>
<td></td>
<td>Fineness module</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Surface cleanliness (P)</td>
<td>79</td>
<td>72</td>
</tr>
<tr>
<td></td>
<td>Sand equivalent (ESV)</td>
<td>0.37</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td>Value to bleu (Vb)</td>
<td>77</td>
<td>53.0</td>
</tr>
<tr>
<td></td>
<td>CaCO₃ content (%)</td>
<td>14.0</td>
<td>08.0</td>
</tr>
<tr>
<td></td>
<td>Coefficient of flattening</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Los Angeles (LA)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Micro deval (MDE)</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 1. Particle size of the different granular classes

Table 6. Chemical analysis of the two sands

<table>
<thead>
<tr>
<th>Sand</th>
<th>SiO₂</th>
<th>Fe₂O₃</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>SO₃</th>
<th>NaCl</th>
<th>Fire loss</th>
</tr>
</thead>
<tbody>
<tr>
<td>ENG</td>
<td>0.06</td>
<td>0.06</td>
<td>0.04</td>
<td>55.80</td>
<td>0.27</td>
<td>0.00</td>
<td>0.35</td>
<td>42.00</td>
</tr>
<tr>
<td>SCIMAT</td>
<td>2.64</td>
<td>0.80</td>
<td>0.35</td>
<td>37.00</td>
<td>6.96</td>
<td>0.00</td>
<td>0.76</td>
<td>46.50</td>
</tr>
</tbody>
</table>

Physical, morphological and mechanical properties are shown in Table 5.

Two sands of the same mineralogical nature, but coming
from two different sites, were used. The first sand rated S-ENG from the giant ENG (National Aggregates Company) quarry in Ain Touta (NA 5043 Standard: Article 10), made up of 98% clean limestone. The second sand is that of the SCIMAT noted S-SCIMAT, consisting of 96% limestone, 0.28% sulphates and 0.32% chlorides. Their chemical compositions are given in Table 6.

We have grouped together in Figure 1 the granulometric curves of these different aggregates in the raw state (non-defillerized sands). Note that S-ENG sand contains 15.8% crushing fines and S-SCIMAT sand a percentage of 16.7%.

2.2.4 Fillers
We chose a quarry limestone filler with a specific Blaine surface area of 375.97 m²/kg, and very little active (activity index of 0.5). This filler consists of calcium carbonate (Table 7).

Table 7. Chemical analysis of the filler

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Filler (%)</th>
<th>Al₂O₃ (%)</th>
<th>CaCO₃ (%)</th>
<th>MgCl (%)</th>
<th>NaCl (%)</th>
<th>Methylene Blue Value (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeO₃</td>
<td>0.02</td>
<td>0.09</td>
<td>98.50</td>
<td>0.35</td>
<td>0.58</td>
<td>0.58</td>
</tr>
</tbody>
</table>

2.2.5 Sand
We added variable filler contents to previously washed and sieved sand at 80 μm and studied the changes in the fineness modulus and sand equivalent as a function of the filler content.

In order to study the evolution of the grain size and the fineness modulus of the two sands, a preliminary study was carried out.

Figure 2. Variation of the fineness modulus as a function of the percentage of filler

(1) Evolution of the fineness modulus according to the percentage of filler. The fineness modulus decreases as the fines content increases, with differences between the two sands (Figure 2). The sand S-SCIMAT is quite coarse with a fineness modulus equal to 3.2; the addition of filler improves it significantly. The sand S-ENG with added filler retains a preferential fineness modulus of between 2.3 and 2.6.

(2) Evolution of the sand equivalent as a function of the percentage of filler. Figure 3 shows that the equivalent of sand measured by the plunger decreases as the filler content increases, but remains above 65 in the range 0 to 12% fines for both sands.

We noted that the values obtained in the methylene blue « VB » test remain constant regardless of the amount of filler introduced.

Figure 3. Variation of sand equivalent as a function of the percentage of filler

2.3 Methodology test
Nine mixtures of thirty-six concrete test pieces prepared in this experimental work according to the reconstituted sand (Sand + filler %) using Portland CEM I cement with a characteristic 28-day strength of 50.3 Mpa.

All mixtures were manufactured according to XP P 18-545, with a binder content of 350 kg and fixed aggregate content of class 4/8 of 195 kg, class 8/16 of 373 kg and class 16/25 of 628 kg.

The limestone fillers were mixed with the two sands S-ENG and S-SCIMAT with a rate ranging from 6 to 24%, with a step of 6%.

An additional amount of water or less may be substituted for certain compositions to provide sufficient workability.

The proportions by mass of the different constituents of the concrete are mentioned in Table 8.

Table 8. Identification of different mixtures

<table>
<thead>
<tr>
<th>Concrete type</th>
<th>Constituents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regular concrete BO</td>
<td>350</td>
</tr>
<tr>
<td>BOS-SCIMAT-6F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-SCIMAT-12F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-SCIMAT-18F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-SCIMAT-24F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-ENG-6F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-ENG-12F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-ENG-18F</td>
<td>350</td>
</tr>
<tr>
<td>BOS-ENG-24F</td>
<td>350</td>
</tr>
</tbody>
</table>
To determine the influence of the W/C ratio, fresh concrete is put on a vibrating table to determine its consistency.

In the anhydrous state, concrete has undergone chemical treatments to determine density and solid mass tests according to EN 196, NF EN 196-1.

The mechanical properties studied in prepared concrete are compression strength on normalized cylinder test pieces (16 x 32 cm) following the NF P-18-406 standard and bending traction on prismatic test pieces (7 x 7 28 cm) following the NF P-18-400 standard.

After pulling at 24 hours of age, the test pieces are kept in a wet environment (20°C and 100% RH) until the test age is 7, 28 and 90 days according to the NF P15-402 standard.

3. RESULTS AND DISCUSSION

3.1 Influence of fillers in workability

3.1.1 Report W/C

We have obtained significantly constant cone thicknesses (9 ± 1 cm) with W/C ratios ranging from 0.52 to 0.62, corresponding to variations in water levels between 3 and 16 liters/m3 of concrete (Figure 4).

The water intake is an increasing function of the frying assay up to 18% of the frying dosing and then decreases further. We can explain this by the fact that the fillers have a very large specific surface, which forms a colloidal microstructure that holds the water. This finding remains variable regardless of the mineral nature of the sand. Beyond 18% thin, fillers behave like a fluidizer, but do not give concrete a notable segregation.

3.1.2 Solid mass of fresh concrete

The density gives an inaccurate indication of the quantitative aspect of porosity and does not give any information about the qualitative aspect of porosity; this is useful for finding possible relations with the mechanical characteristics of the material.

The fine assay, up to 18%, increases the density of the material. Solid masses are quite low, probably reflecting high porosity, and vary between 2.25 and 2.40 t/m3; the optimal compactness is achieved at 18% fine. However, for mixtures containing more than 18% fine, we get a drop in the density of the solid masses, in the order of 2% for BOS-ENG concrete and 6% for BOS-SCIMAT concrete.

These findings allow us to conclude that fine limestone:

- Change the geology of fresh concrete while performing a bridging role of porosity (more compact granular stacking).
- Sometimes have a positive, sometimes negative influence on density based on their content (Figure 5).

3.1.3 Percentage of air entrapped

The use of fillers up to 18% results in a reduction of 1 to 1.5% of the air content covered, but the content again increases only for S-SCIMAT sand beyond 18% of the frying content (Figure 6).

3.2 Influence of fillers in the resistance

3.2.1 Mechanical performances

The results of the mechanical resistances of the two concretes hardened over time given in Figures 7 and 8 show that these resistances increase with the percentage of filler and pass through an extremum for approximately 18% of filler. These results are reflected in the fact that the mixtures become more compact when we add fillers up to around 18%.

Beyond 18% filler, the evolution of the resistances is reversed and even evolves more quickly: with 24% filler.

We also notice that the highest compressive and tensile strengths are obtained with BOS-SCIMAT concretes.

Although the influence of fillers is a little less strong in tension by bending than in compression, it seems that the presence of fines in the aggregates risks above all being the cause of drops in tensile strength.
3.2.2 Physical performance

The curves obtained (Figures 9 and 10) show that the fines dosage increases the shrinkage of the concrete, which can reach values three times that of a concrete without fines. This result is probably related to a different distribution of sizes and voids between the two materials.

The weight evolution of the specimens presents the same pace as the shrinkage, although in a less accentuated way, the drying shrinkage being preponderant compared to the hydration shrinkage.

It seems that the action of fillers on shrinkage is twofold; on the one hand we have a water retention effect which slows down shrinkage at short maturities; on the other hand, we have a general effect due to the activity of the fines which systematically increases the shrinkage.

**Figure 7.** Compressive strength of reconstituted sand concretes at the age of 7, 28, and 90 days

**Figure 8.** Tensile strength of reconstituted sand concretes at the age of 7, 28 and 90 days
Figure 9. Removal of reconstituted sand concrete at 7, 28, and 90 days of age

Figure 10. Mass loss of concretes with reconstituted sand at the age of 7, 28, and 90 days

4. CONCLUSIONS

This article presents undeniable interests both in terms of the exploitation of regional resources and in terms of the environment and the economy.

Indeed, the study undertaken in the latter, indicates to us that it is possible to exploit the fine limestones in the concretes made by introducing various rates up to 24% by substitution with the sand which were compared in order to valorize sands with high levels of fillers. These concretes have been formulated maintaining a constant workability. The rheological, physical and mechanical properties have been studied using various experimental techniques.

Our results show that with concretes based on aggregates from limestone crushing, the presence of a certain content of fines in the sand is systematically beneficial from the point of view of mechanical performance.

Concrete without filler gives significantly lower strengths than concrete with filler.

At the end of this work, we can try to give a number of conclusions on the beneficial actions provided by limestone fillers:

- The presence of fillers in calcareous sands is a necessity, because they not only improve the rheological properties, but also the mechanical and physical performance of the concretes.
- A thorough washing of the aggregates goes in the opposite direction of the improvement of the characteristics of the concrete.
- The optimum filler content which allows the highest mechanical strengths to be obtained, measured on concrete specimens of constant workability, is around 18% fines, regardless of the origin of the aggregate. Beyond the rate of 18% of fines, the demand for water decreases and the fines begin to behave as a water-reducing adjuvant. The cement-filler-aggregate interaction causes a recovery of the occluded air content above 18% of fines. Strength gains are linked to the improvement in compactness obtained by adding fines, and the drop-in strength (beyond 18% filler) is mainly due to this increase in occluded air.
- The tensile strengths are less influenced by the dosage of fillers, which moreover goes against generally accepted ideas.

Recommendations for future studies, in particular, an in-depth physic-chemical and microstructural study on permeability, which is an indicator of durability, as well as the
shrinkage and freeze-thaw behavior of concretes containing high filler contents.

REFERENCES


