Thermal and mechanical behavior of recycled concrete aggregates and subjected to high temperatures

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REFERENCE:

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PREMESSA

L’elaborato di tesi scaturisce dalla collaborazione con l’Università francese di Cergy-Pontoise durante il mio soggiorno Erasmus semestrale; nel periodo ho preso parte al progetto di studio sul calcestruzzo riciclato oggetto di tesi di dottorato della dottoranda Cléo Laneyrie.

La ricerca è finalizzata ad individuare la possibilità di utilizzare gli aggregati riciclati per sostituire parzialmente l’aggregato naturale a seguito di:

- costi per smaltimento in discarica di materiale di demolizione sempre maggiori;
- limitazioni imposte da trattati per il rispetto dell’ambiente;
- scarsità e difficoltà del reperimento di risorse naturali;
- soddisfare la richiesta sul mercato di nuovi prodotti specifici per la costruzione.

La ricerca si basa su un'analisi sperimentale con quattro tipologie di calcestruzzo differenti: due analizzano gli aggregati grossi riciclati mentre due riguardano la diversificazione del rapporto acqua/cemento.

L’obiettivo del mio lavoro consiste nell’analizzare l’influenza di un tipo di aggregato riciclato (BRM 0,3) sul comportamento termo-idro-mecanico del calcestruzzo sottoposto ad alte temperature.

La prima parte dello studio verte sulla ricerca bibliografica al fine di analizzare in dettaglio tutte le caratteristiche e i problemi che presenta l'aggregato sottoposto a verifica. La seconda parte è dedicata alla formulazione e attuazione del calcestruzzo riciclato. È stato prodotto calcestruzzo riciclato attraverso l'utilizzo di aggregato grosso derivato da un calcestruzzo "genitore" gettato in laboratorio di cui sono note tutte le caratteristiche. Gli aggregati grossi riciclati sono mescolati con ordinario aggregato fine, per garantire la qualità richiesta del calcestruzzo strutturale. Essendo le proprietà degli inerti riciclati diversi da quelli naturali, sono esposte le caratteristiche dell’aggregato utilizzato. Sono state inoltre illustrate le proprietà del calcestruzzo fresco e indurito.

L’ultima parte si concentra sui risultati ottenuti dalle varie prove. Il calcestruzzo in esame subisce cicli di riscaldamento/raffreddamento ad una velocità di 0,5°C/min a temperature di 150°C, 300°C, 450°C e 750°C, come richiesto dalla norma. Saranno poi determinate l'evoluzione della fessurazione, le proprietà meccaniche, fisiche e termiche.
INTRODUCTION

The thesis comes from the collaboration with the French University of Cergy-Pontoise during my stay Erasmus of six months; during the period I took part in the study project on recycled concrete object of the PhD thesis of Cléa Laneyrie.

Concrete, it has been claimed, is not an environmentally friendly material due to its destructive resource-consumption nature and severe environmental impact after its use. Nevertheless, it will remain one of the major construction materials being utilized worldwide. Taking the concept of sustainable development into consideration, the concrete industry has to implement a variety of strategies with regards to future concrete use, for instance; improvements in the durability of concrete and the better use of recycled materials. It is estimated that the annual generation of construction and demolition waste in the European Union could be as much as 450 million ton, which is the largest single waste stream, apart from farm waste. Such large consumption of natural aggregates will cause destruction of the environment.

Owing to the increasing cost of landfill, the scarcity of natural resources coupled with the increase in aggregate requirement for construction, the use of recycled aggregate to partially replace the natural aggregate has, therefore, become more common. Indeed, in some European countries, a large proportion of aggregate comes from secondary sources. Most European Union member countries have established goals for recycling that range from 50% to 90% of their construction and demolition waste production, in order to substitute natural resources such as timber, steel and quarry materials. Recycled materials are generally less expensive than natural materials, and recycling in Germany, Holland and Denmark is less costly than disposal.

Even though the utilization of recycled aggregates in the concrete industry has been taking place for many years, the promotion of this recycled material as an alternative has never been easy in the industry. Basically, recycled aggregates are seldom utilized in structural constructions, instead they have been used as fillers in road construction and in low-level applications due to material defects such as large water absorption
capacity and their elongated and angular shape. Currently, 6% of aggregate products in France are recycled (21M out of 376M).

Furthermore, the utilization of recycled aggregates might at least lead the concrete industry to embrace the concept of sustainable development in the near future.

The research is based on an experimental analysis with four different types of concrete: two analyze the recycled coarse aggregates each with two different water/cement ratios.

The goal of my work is to analyze the influence of a type of recycled aggregate (BRM 0,3) on the thermo-hydro-mechanical behavior of concrete subjected to high temperatures. This study shows for first a literature search in order to analyze in detail all the features and problems that presents the aggregate chosen. The second part is devoted to the formulation and implementation of the recycled concrete. It has been produced recycled concrete through the use of coarse aggregate from a "parent" concrete cast in laboratory that we known all features. The recycled coarse aggregates are mixed with ordinary fine aggregate, to ensure the quality required of structural concrete. As the properties of recycled aggregates are different from those of natural aggregates, the characteristics of recycled coarse aggregate used will be presented. The properties of both fresh and hardened concrete will be illustrated. The initial water content is very important on the properties of fresh and also hardened concrete.

The last part focuses on the results obtained from the various tests. This concrete undergo the heating/cooling cycles at a rate of 0,5°C/min to temperature of 150°C, 300°C, 450°C and 750°C, as required by the norm. We determine the evolution of the cracking, the thermal properties, the physical and mechanical properties of the concrete with the temperatures experienced. The residual mechanical behavior of concrete varies with the nature of aggregates and the influence of aggregate depends also on the compactness of the cement paste. The recycled aggregate present also an instability because of the decarbonation/hydration phenomena but after the heating/cooling cycle of 750°C. The initial water content has also an importance on the thermal behavior of recycled aggregates.
CHAPTER 1 - BIBLIOGRAPHY

1.1 GENERAL INFORMATION ON THE CHARACTERISTICS OF CONCRETE

Concrete is a composite material having a highly heterogeneous mixture consisting of aggregates, a cement paste optionally incorporating additives and admixtures. Concrete can also be considered as a multiphase material containing three phases: solid (aggregate and cement paste), liquid (free and adsorbed water) and gas (air and water vapor). Its mechanical properties are developed through the hydration of cement. The complexity of its microstructure is one of the causes of the particular mechanical behavior when subjected to various stresses (heat, water, mechanical, chemical ...). Therefore it is necessary to know the micro structure and the behavior of each of the phases presented in order to understand in the macroscopic scale the response of the concrete’s surface that they may occur, when it solicited.

1.1.1. The aggregate

Aggregates occupy a large part of the concrete volume (55-80%) and are the source of its strength [Xing 2011]. It is an inert material at room temperature. The aggregates used must have good mechanical strength and their size distribution curve must be optimized to fill the possible voids in the concrete. This is the reason why many aggregates are used in the same concrete are sand, grit and gravel. Aggregates are generally of natural origin. They are mainly siliceous and calcareous sedimentary rocks, metamorphic rocks such as quartzite, or igneous rocks such as basalt, granite or porphyry. Regardless of
their nature, can be alluvial aggregates (called rounded aggregates) or career (so crushed aggregates). The origin of the aggregates can also be artificial (of mineral origin, processed, or slag aggregate) or after recycling (e.g. crushed concrete). For concrete with good features, several parameters are involved in the choice of aggregates such as the quality (e.g. mechanical, physico-chemical, cleanliness and so on), mineralogy, form aggregates and appropriate granulometry.

Before specifically address this point, it should be noted that we have deliberately limited the literature studies to recycled aggregates because it is the source of aggregates used to perform our research.

1.1.2. Cement paste

The cement paste is 25 to 40% of the total volume of the concrete. This is the paste that performs the role of matrix within the concrete binder aggregates. It is made by mixing cement and water.

Cement is commonly used in civil engineering cement "Portland" that is a hydraulic binder. It is capable to set and harden with water. In the mixing occurs hydration, that is a hydraulic binder reacts chemically with water.

In case of recycled aggregate concrete it will be necessary to add more cement in concrete made with 100% of recycled aggregate in order to achieve the same workability and compression strength as conventional concrete [Etxe 2007].

1.1.2.1. Composition of the cement

The main component of the cement is clinker, which is obtained from cooking a mixture of lime and suitable clay in a kiln respectively average percentage of 80%-20%. The main constituents of clinker represent over 90% of Portland cement and determine its properties [Baro 1994]. The cement consists essentially of the following four mineralogical constituents:
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- Tricalcium silicate (or Alite) 3CaO, SiO\textsubscript{2} (denoted C\textsubscript{3}S): 60 to 65%
- Dicalcium silicate (or Belite) 2CaO, SiO\textsubscript{2} (denoted C\textsubscript{2}S): 20 to 25%
- Tricalcium aluminate (or Celite) 3CaO, Al\textsubscript{2}O\textsubscript{3} (denoted C\textsubscript{3}A): 8 to 12%
- Tricalcium ferrite aluminate 4CaO, Al\textsubscript{2}O\textsubscript{3}, Fe\textsubscript{2}O\textsubscript{3} (denoted C\textsubscript{4}AF): 8 to 10%

Clinker, once crushed and mixed with a small amount of gypsum (about 5% of the cement), becomes pure Portland cement. The addition of gypsum (CaSO\textsubscript{4}, 2H\textsubscript{2}O) in the cement composition can slow down the setting for the temporary formation of crystals of ettringite (C\textsubscript{3}A, 3CaSO\textsubscript{4}, 32H\textsubscript{2}O) in the C\textsubscript{3}A grain surface is very reactive with water. This slows down the hydration of C\textsubscript{3}A and avoids the phenomenon of rapid setting (rapid stiffening of the dough, the result of a lack of ion SO\textsubscript{4}\textsuperscript{2-} in solution mixing) [Baro 1994].

1.1.2.2. **Hydration of the concrete**

When adding water to the cement hydration reactions are triggered causing the formation of a porous network and the formation of hydrated products. The main hydrates formed are calcium silicate hydrate CSH, portlandite (or calcium hydroxide) Ca(OH)\textsubscript{2}, the calcium aluminate hydrate, ettringite (3CaO, Al\textsubscript{2}O\textsubscript{3}, 3CaSO\textsubscript{4}, 32H\textsubscript{2}O etc.).

The main phase of chemical reaction of hydration of cement is:

\[
\begin{align*}
C_3S & \quad \text{Tricalcium silicate} \\
C_2S & \quad \text{Dicalcium silicate} \\
\end{align*}
\quad + \quad H_2O \quad \xrightarrow{\text{exothermic}} \quad \text{CSH} \quad + \quad Ca(OH)_2 \quad \text{Portlandite}
\]

For a given cement, the amount of CSH and Ca(OH)\textsubscript{2} formed mainly depend on the W/C ratio (water/cement) and reaction time [Diam 2004]. In an ordinary hardened cement paste, there are on average 50 to 70% calcium silicate hydrate CSH and from 25 to 27% of portlandite (calcium hydroxide) Ca(OH)\textsubscript{2} (Figure 1.1). Thus, the CSH is the most important phase of hydration of hardened cement paste. The main mechanical properties of the material will depend from this [Baro 1994]. It is important to note that the amount of CSH is more important in a cement paste with a high-performance
ordinary cement paste [Xing 2011]. For cons, the portlandite Ca(OH)\(_2\) has little significance in terms of strength because its large crystals are likely to limit the compressive strength of concrete. In addition, the portlandite is easily soluble in water, which reduces the durability of concrete [Hage 2004]. Therefore, to reduce the amount of portlandite, can be added to the silica fume cement. Silica fume reacts with portlandite in a pozzolanic reaction.

It consumes the portlandite and form additional CSH (Calcium Silicate Hydrate). Fumed silica, due to its grain size, lower than that of the grains of cement also increases the compactness of the matrix. This leads to improved mechanical performance of concrete.

The use of silica fume, with plasticizers, is widespread in the manufacture of high performance concretes. Thus, in the case of cement pastes with high performance, the amount CSH phase is even more important than in the case of ordinary cement pastes.

![Image](image.jpg)

*Figure 1.1: A hexagonal crystal Portlandite (center) surrounded by CSH gel*

1.1.3. Microstructure of cement paste

The pores in the hardened material has a complex geometry and dimensions vary. The porosity varies from one concrete to another in terms of quality and quantity (pore size and distribution of pore radius). The pore structure and pore distribution within the
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Concrete play a very important role not only on strength, but also on the transport phenomena and consequently on the durability [Baro 1994].

Two families of pores are distinguished: the pores on the capillaries and pores related to hydrates.

- The capillary pores: these are the remains of the intergranular spaces of fresh dough. They may or may not be filled with water and the volume decreases during hydration for a particular ratio of water / cement [Bill 2003].

- The intrinsic porosity to CSH: the porosity in scale even smaller. Its characteristic dimension of nanometer, is much smaller than the capillary porosity. The representations that are made differ, in general, two types of pores [Baro 1994].
  1. Pores inter-crystallites (inter-lamellar space) which are located between the gel particles.
  2. Intra-crystallite pores (inter-area sheets) which are located within the same gel particles, it is extremely small spaces.

1.1.4. Water content in the cement paste

The water, as well as cement and aggregates, one of the constituents of concrete. It operates, in the mechanical and physico-chemical properties, at all stages of the life of the material. It gives concrete plasticity that allows the workability. It then provides the cement hydration and contributes to the cohesion of the cured material. Water is generally classified according to the nature of his relationship with the hydrated cement paste. The classification used in accordance with order of bonds increasing [Xing 2011].

- Free and capillary water: it consists of the condensed phase is not within the scope of influence of surface forces. It is mainly found in the capillary pores larger than 10 microns (large pores and cracks) dimension. This water is the first to migrate and escape during evaporation that occurs between 30 and 120 ° C.
• The adsorbed water: This is water molecular layers on the solid surface of the pores.

The adsorbed water is subjected to fields of surface forces from the solid.

• The combined water: it is a component of cement hydrates as water of solvation and water of crystallization. Two types of structure can be given as an example: ettringite and CSH.

1.1.5. Bonding cement paste – aggregates

The mechanical properties of concrete and their resistance to aggressive agents depend on those of the matrix of hydrated cement, the nature and the granulometry of the coated elements, the proportions of each aggregate, and their spatial configuration. But they are also a function of the bond that develops between these two phases. Provided that the aggregate is clean, the adhesion is sufficient to ensure the continuity matrix-coated materials. But it is not enough that the continuity between the two phases is ensured. The connection may be the weak point of the association vis-à-vis mechanical stress condition and then the strength of the assembly.

Microstructural investigations helped to highlight the existence of a particular area of hydrated paste around aggregates in hardened concrete. This area is called the interfacial zones. It is characterized by the presence of large pores where there are large crystals. The connection which is established during the hydration between the cement paste and coated materials it depends on the nature of the two phases in contact, the concentration of cement paste and the storage conditions.

By optical microscope observations and analysis by X-ray diffraction, Farran (1956) showed that the nature of the compounds of the transition zone, and the quality of the bonding cement paste/aggregate, depend essentially on the mineralogical aggregates and, of course, that of cement.

In concrete, the interfacial zone between cement paste and aggregate plays a critical role. At the macroscopic level, concrete is a composite material consisting of discrete
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aggregates dispersed in a continuous cement paste matrix [Bentz 2003]. As with other composites, the bond between these two major components of concrete critically determines the mechanical performance. The interfacial zone is the weak link of concrete and therefore, when the concrete is subject to certain constraints, the cracks begin to develop in the transition zone [Li 2001].

In fact, the structure of recycled aggregate concrete is much more complicated than that in normal concrete. Recycled aggregate concrete possesses two interfacial zones, one between the recycled aggregate and new cement paste (new interfacial zone) and the other between the recycled aggregate and the old mortar attached (old interfacial zone), which are schematically shown in Figure 1.2. The cement mortar remains at the interfacial zone of recycled aggregate form the weak link in recycled aggregate concrete, which is composed of many minute pores and cracks, and they critically affect the ultimate strength of the recycled aggregate concrete. These pores and cracks increase consumption of water leading to less water for hydration at the interfacial zone of recycled aggregate concrete [Vivi 2004].

![Figure 1.2: Interfaces of recycled aggregate.](image)

Under the examination of scanning electron microscopy, the cracks within recycled aggregate remain unfilled (Figure 1.3) [Vivi 2004].
The quality of interfacial zone depends on surface characteristics of the aggregate particles, the degree of bleeding, chemical bonding and the specimen preparation technique which, however, are notoriously difficult to measure. Although these effects have been reported by some investigations, the results are difficult to reconcile. Nonetheless, it is generally agreed that as the paste-aggregate bond strength increases, the concrete strength also increases [Mind 2003].

The bond strength also increases with the age of the concrete as it depends as much on the strength of the hydrated cement paste that surface properties of the aggregate. The bond strength and the strength of the hydrated cement paste may increase with age, so that no cracking begins interface paste/aggregate. This is no longer the strength of the bond that controls the strength of concrete.
1.2. PROPERTIES OF RECYCLED AGGREGATES

1.2.1 Aggregate production plant

Recycled concrete aggregate could be produced from:

- recycled precast elements and cubes after testing,
- demolished concrete buildings.

Whereas in the former case, the aggregate could be relatively clean, with only the cement paste adhering to it, in the latter case the aggregate could be contaminated with salts, bricks and tiles, sand and dust, timber, plastics, cardboard and paper, and metals. It has been shown that contaminated aggregate after separation from other waste, and sieving, can be used as a substitute for natural coarse aggregates in concrete [Naga 2004].

The equipment, used in most of the studies found, simply consists of a series of successive crushers, with oversize particles being returned to the respective crusher to achieve desirable grading, as is show in Figure 1.4. The best particle distribution shape is usually achieved by primary crushing and then secondary crushing, but from an economic point of view, a single crushing process is usually most effective. Primary crushing usually reduces the waste concrete rubble to about 50mm pieces and on the way to the second crusher, electromagnets are used to remove any metal impurities in the material [Cori 2002]. The second crusher is then used to reduce the material further to a particle size of about 14–20 mm. Care should be taken when crushing brick material because more fines are produced during the crushing process than during the crushing of concrete or primary aggregates.
As with natural aggregate, the quality of recycled aggregates, in terms of size distribution, absorption, abrasion, etc. also needs to be assessed before using the aggregate.

1.2.2. The recycled coarse aggregates

Acceptable properties of aggregates are an elemental base for concrete quality, however adequate mix proportions and concrete production methods are highly important in concrete quality too. Recycled aggregates are composed of original aggregates and adhered mortar. The physical properties of recycled aggregates depend on both adhered mortar quality and the amount of adhered mortar. The adhered mortar is a porous material, its porosity depends upon the w/c ratio of the recycled concrete employed [Naga 2000]. The crushing procedure and the dimension of the recycled aggregate have an influence on the amount of adhered mortar [Etxe 2007]. The density and absorption
capacity of recycled aggregates are affected by adhered mortar and they must be known prior to the utilization of recycled aggregates in concrete production in order to control properties of fresh and hardened concrete. The increased absorption of recycled aggregate, means that concrete made with recycled coarse aggregates and natural sand typically needs 5% more water than conventional concrete in order to obtain the same workability. Some researchers suggest a limit of 30% of recycled aggregate in order to maintain the standard requirements of 5% of absorption capacity of aggregates for structural concrete [EHE 1999].

In general the workability of recycled aggregate concretes is affected by the absorption capacity of the recycled aggregates. The shape and texture of the aggregates can also affect the workability of the mentioned concretes. This depends on which type of crusher is used [Shok 1997].

With respect to compressive strength, concrete made with 100% of recycled coarse aggregate with lower w/c ratio than the conventional concrete can have a larger compression strength. When the w/c ratio is the same the compression strength of concrete made with 100% of recycled aggregate is lower than that on conventional concrete [Tava 1996].

The employment of different qualities of recycled aggregate in concrete production brings about an increase in the compressive strength variation coefficient [Etxe 2007]. Any variation in concrete production or in the properties of the constituents used produces a variation of strength in the resultant concrete.

1.2.3. The natural fine aggregates

The utilization of recycled sand was avoided, due to its absorption capacity, which would no doubt produce a shrinkage effect [Etxe 2007]. The quantity of adhered mortar increases with the decrease of size of the recycled aggregates [Hans 1992]. Therefore in a lot of research, limestone sand have been used as fine aggregate in all concrete mixes.
1.2.4. Absorption

The absorption capacity is one of the most significant properties which distinguishes recycled aggregate from raw aggregates, and it can have an influence both on fresh and hardened concrete properties. The water absorption in recycled aggregates ranges from 3 to 12% for the coarse and the fine fractions [Jose 2002] [Katz 2003] [Rao 2005] with the actual value depending upon the type of concrete used for producing the aggregate. It may be noted that this value is much higher than that of the natural aggregates whose absorption is about 0.5–1%. The high porosity of the recycled aggregates can mainly be attributed to the residue of mortar adhering to the original aggregate. This, in fact, also affects the workability and other properties of the new concrete mix.

If recycled aggregates are employed in dry conditions the concrete's workability is greatly reduced due to their absorption capacity. Some researchers argue that the recycled aggregates should be saturated before use [Neal 1998]. If the recycled coarse aggregate is not humid, it would absorb water from the paste thus losing both its workability in the fresh concrete, and also the control of the effective w/c ratio in the paste.

A recommended level of humidity could be 80% of the total absorption capacity, however the most important factor is that the aggregates employed are wet in order to reduce their absorption capacity. In this case the mechanism could be that recycled aggregate that had a moderate initial moisture content absorbed a certain amount of free water and lowered the initial w/c in the interfacial transition zone at early hydration. Newly formed hydrates gradually filled the region processes effectively improved the interfacial bond between the aggregates and cement [Poon 2004]. One should note, however, that the recycled aggregates should not be saturated, as that would probably result in the failure of an effective interfacial transition zone between the saturated recycled coarse aggregates and the new cement paste. Barra de Olivera and Vázquez [Barra 1998], note a slight decrease especially in flexural strength of the concrete made from saturated recycled aggregates. In this study, for reasons of workability, recycled coarse aggregates were put in water for 24h before their use and were put it to drain in order to obtain the saturated-surface-dry.
The fresh concretes were made with 12-16 cm slump. Since we want the same workability of conventional concrete, was not changed the amount of superplasticizer due of recycled aggregates that were saturated with dry surface (normally recycled aggregate concretes have less workability). The workability of fresh concrete was determined by a slump test, in accordance with NF EN 206-1
1.3. MICROSTRUCTURAL CHANGES OF THE CEMENT MATRIX UNDER THE EFFECT OF TEMPERATURE

Concrete is a poor conductor of heat, but can suffer considerable damage when exposed to fire. Unraveling the heating history of concrete is important to forensic research or to determine whether a fire-exposed concrete structure and its components are still structurally sound. Assessment of fire-damage concrete structures usually starts with visual observation of color change, cracking and spalling. On heating, a change in color from normal to pink is often observed and this is useful since it coincides with the onset of significant loss of concrete strength.

Of particular importance are loss in compressive strength, cracking and spalling of concrete, destruction of the bond between the cement paste and the aggregates and the gradual deterioration of the hardened cement paste. Also, differing thermal expansion between aggregates and cement paste can create surface crazing, which can lead to deeper cracking [Geor 2005].

The main physico-chemical reactions in the cement matrix during its heating are summarized in Table 1.1 [Noum 1995]. These reactions are characterized by endothermic and exothermic peaks in the differential temperature curves of differential thermal analysis DTA.
<table>
<thead>
<tr>
<th>Temperature range</th>
<th>Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>30-120°C</td>
<td>The evaporable water and a part of the bound water escapes. It is generally considered that the evaporable water is completely eliminated at 120 °C.</td>
</tr>
<tr>
<td>130-170°C</td>
<td>The decomposition of gypsum (with a double endothermal reaction) [Noum 1995], the decomposition of ettringite [Zhou 2001] and the loss of water from part of the carboaluminate hydrates [Noum 1999] take place.</td>
</tr>
<tr>
<td>180-300°C</td>
<td>The heat breaks the cement gel and hard water molecules in hydrated silicates. The chemically bound water begins to escape from the concrete [Noum 1995].</td>
</tr>
<tr>
<td>At 250 and 370 °C</td>
<td>There may be small endothermic peaks indicating the effects of decomposition and oxidation of metallic elements (ferric) [Noum 1995].</td>
</tr>
<tr>
<td>450-550°C</td>
<td>Dehydroxylation of the portlandite (calcium hydroxyde) [Noum 1995] and [Platr 2002].</td>
</tr>
<tr>
<td>600-700°C</td>
<td>There decomposition of CSH phases and formation of β-C2S. This is the second stage of dehydration of calcium silicate hydrates, which produces a new form of silicates bicalcium [Noum 1995].</td>
</tr>
<tr>
<td>Over 1300-1400°C</td>
<td>Beginning of the merger of the cement paste.</td>
</tr>
</tbody>
</table>

Table 1.1: The main physico-chemical reactions in concrete at high temperature

It is now clear that these changes are influenced by a considerable number of environmental factors, among which the temperature level, the heating rate, the stress state and the external sealing and moisture content, the latter having also a remarkable influence on explosive spalling. The mix design, namely the type of aggregate and
cement and the interaction between them has also a major influence on the way concrete degrades with temperature [Khou 2000].

From Figure 1.5, we observe that the disappearance of ettringite occurs before 100°C with a gradual dehydration of CSH to 600°C. The amount of CaO increases sharply from 500°C, due to dehydration of portlandite [Cast 2004].

![Figure 1.5: Evolution de la quantité des phases de la pâte de ciment portland au cours d'un traitement thermique [Cast 2004].](image)

### 1.3.1. Post-fire mechanical performance of recycled concrete

As aggregates occupy 60-80% of the volume of concrete, variations of their thermal properties have a significant effect on the performance of concrete at a high temperature. The thermal deformation and the thermal conductivity of the concrete are significantly influenced by the conduct of recycled aggregates. The physico-chemical changes also occur in recycled aggregate since they are composed of natural ones, according to their mineralogical nature.

Very few studies are reported in the literature concerning the behaviour of recycled concrete at very high temperatures.
One of the few studies [Zega 2006], compares the post-fire compressive strength and the elasticity modulus of conventional concrete made with natural coarse aggregate (granitic crushed stone), comprising different water/cement ratios (0.40, 0.55 and 0.70), with recycled concrete of similar characteristics, incorporating 75% by volume of recycled aggregates. These latter aggregates were obtained from crushing waste concrete produced with granitic crushed stone. Cylindrical specimens (150×300 mm) were heated in an electric oven, according to a non-standardised heating curve, up to a temperature of 500 °C for periods of around 1 h and 4 h and then cooled down slowly.

Although, in general, the post-fire performance of recycled concrete and conventional concrete was similar, in terms of both compressive strength and elasticity modulus, for a w/c ratio of 0.40 and 1 h of thermal exposure, the recycled concrete presented better mechanical performance than the conventional concrete.

Other similar studies were performed from the same authors, but with a concrete produced with natural and recycled coarse aggregates of different origin, namely granitic crushed stone, siliceous gravel and quartzitic crushed stone. In these experiments, the authors confirmed the better post-fire mechanical performance of recycled concrete, particularly for the lowest w/c ratio (0.40) and when produced with quartzitic aggregates [Zega 2009].

Xiao and Zhang [Xiao 2007] evaluated the residual compressive strength of recycled concrete with different replacement rates (0%, 30%, 50%, 70% and 100%) of natural siliceous coarse aggregates by recycled siliceous coarse concrete aggregates, obtained from the demolition of an abandoned airport runaway. Cubic specimens (150 mm) were heated according to ISO 834 standard curve up to predefined temperature levels (200 °C to 800 C, in steps of 100 C), which were kept constant for over 2 h and, finally, the oven door was opened and the specimens were cooled down to room temperature inside the oven. The authors concluded that for a replacement rate of 30% the residual compressive strength of recycled concrete was lower than that of conventional concrete. However, for replacement rates not less than 50% the residual compressive strength of recycled concrete became considerably higher than that of RC. In addition, for these higher replacement rates the residual compressive strength noticeably increased for temperatures between 300 °C and 500 °C.
These results can be attributed to the similar coefficients of thermal expansion between the aggregates and the mortar at the interface of recycled concrete, which would reduce the significance of micro- and macro-cracks during the compression process [Zega 2006].

At high temperatures, the aggregates break down and undergo significant physical, chemical and mineralogical changes that materially alter the microstructural characteristics of the material. Geometry and density of cracks and pores are the main control parameters for the physical properties of recycled aggregates. In recycled aggregate structures, the temperature variation is one of the main factors influencing the integrity and physical properties of recycled aggregates. She is responsible for the evolution of the microstructure of the recycled aggregate by inducing new cracks and development of microcracks and thus increasing the volume of empty space. It is very important to evaluate the thermally induced microcracks at different temperatures.

We therefore investigated the residual post-fire mechanical properties of recycled concrete, namely the compressive strength, the splitting tensile strength (not addressed in the above mentioned investigations) and the elasticity modulus; finally, we analyzed macroscopically, by electron microscopy, the cracks within of recycled aggregate post-fire.
1.4. THE EVOLUTION OF THE PHYSICAL AND MECHANICAL PROPERTIES OF CONCRETE SUBJECTED TO HIGH TEMPERATURE

1.4.1. Evolution of the physical properties of concrete subjected to high temperature

1.4.1.1. Evolution of the mass loss

While heating, the concrete looses weight because of the departure of the free water in capillaries and the bound water which is contained in the hydrates. Khoury [Khou 2000] showed the evolution of the mass loss of concrete while heating (Figure 1.6).

![Graph showing mass loss and rate of weight loss as a function of temperature](image)

*Figure 1.6: Mass loss during the heat and speed of mass loss as a function of the temperature of the concrete.*

The curves of mass loss of the concrete are shown in Figure 1.7, in addition they depend on the temperature, the mineralogical nature of the aggregate influence and the loss of mass of concrete [Xing 2011].
Xing has used two types of matrix (normal or high performance). The paste volume has been kept unchanged for the three natures of aggregates (S=siliceous, SC=silico-calcareous and C=calcareous), so that the difference in mass loss between concretes is only linked to the nature of the aggregate.

With regard to normal concrete the mass loss of different concretes is very similar up to 300°C. Respective to high performance concrete differences between mass loss already appear at 300°C, which can be explained by the free water brought by the aggregates [Xing 2011].

![Figure 1.7: Mass loss of the tested concretes as a function of the heating temperature [Xing 2011].](image)

Hager [Hage 2004] notes that the mass loss recorded during a heating cycle, with all concrete, starts above 100°C. Up to 150°C the mass loss of high performance concretes is slow. It increases between 150°C and 200°C. The kinetics of mass loss increases only sharply from 200°C.

The water evaporates in the material migrates to the inside and the outside of the specimen. Water vapor which migrates toward the cold region and gradually condenses to liquid water fills the pores. The said plug consisting of liquid water prevents the passage of air and water vapor it gradually increases the pressure in the pores. The plug formation occurs more rapidly in high performance concrete due to of their low porosity and permeability.
For the same aggregate, the loss of mass of concrete is significantly higher when the W/C ratio varies from 0.3 to 0.6 [Pliy 2010].

There has not been found any research regarding the loss of mass of the recycled concrete in concerning the heating temperature.

1.4.1.2. Porosity evolution

Concrete is a material porous bi-phase, composed of a solid phase and a porous phase. The total porosity can be measured by saturating water capillary porosity and is generally measured by mercury porosimetry. Research on the evolution of this parameter ([Kali 1998] [Noum 1995]) show that the porosity increases with the temperature. This increase depends partially on the increase in the total volume and the pore size and the micro-cracks caused by the differential expansion between the paste and aggregates.

The national project BHP 2000 [Irex 2005] found that the water porosity changes just a little with temperature. The small variation is attributed to the loss of water located between the layers of hydrates. Drying shrinkage of the sheets the volume of the space freed by the water loss is being reduced. The increase of water porosity is more pronounced from 250 °C due to micro-cracking [Ye 2007].

For cons, the porosity determined by mercury intrusion technique gradually increases when the heat treatment temperature increases from 105 °C to 400 °C. Furthermore, the porosity determined by the mercury intrusion technique are systematically higher than those obtained by the technique of water. This is because mercury can seep into the smallest pores, but we will use the method of water. In addition, the difference is more marked for high performance concretes as for ordinary concrete [Baro 2000].

It is important to know the initial moisture condition of the concrete for porosity [Lau 2006].

We have not found any studies that analyze the evolution of porosity for recycled concrete subjected to high temperatures.
1.4.1.3. Evolution of permeability

The pore space of porous materials strongly affects both mechanical and transport properties. Regarding building material such as concrete, which may be exposed to severe environmental conditions, these properties determine the service life of concrete and concrete structures. Recently, the gas permeability of concrete was identified as the key parameter which controls the explosive spalling and, thus, the integrity of the concrete structures under fire loading.

The durability of concrete is strongly affected by its pore structure, the latter allowing the transport of various substances (e.g., water, vapor, aggressive agents) and therefore determining the life span of structures especially when subjected to aggressive environments (e.g., fire loading, freeze/thaw, sea water, acid attack etc.). When concrete is subjected to fire loading, the transport of water and vapor strongly affects the risk of explosive spalling, caused by the pore-pressure build-up in consequence of evaporation of capillary water [Matt 2008].

Permeability to gas is preferred in comparison to the permeability of water. Concerning water, there is a possibility of migration of fine elements in the smallest pores and also of rehydration of unhydrated cement, resulting in potential autogenous healing of cracked concrete, influenced, furthermore, by temperature [Rein 2003].

Regarding the influence of the recycled aggregates on durability properties, there is few data that has been published. On the shrinkage parameter, Wang et al. [Wang 2001] found out that the increase of the recycled coarse aggregate content in concrete increase the shrinkage as well. A total of 50% increment on shrinkage is registered by the concrete when the coarse aggregate is completely superseded with the recycled coarse aggregates. For air permeability, Sun et al. [Sun 2006] discovered that the permeability of concrete containing 60% of the recycled coarse aggregates that have increased drastically by 196% compared to normal concrete. Olorunsogo and Padayachee [Olor 2002] also concluded that the concrete with 100% recycled aggregates as the coarse aggregate have been graded as “poor” in the oxygen permeability index. This phenomenon is mainly due to the cracks and fissures created within the micro and macro structures of the RCA during the crushing process. However, the permeability of the recycled concrete is possible to be refined through a longer curing duration.
Apart from that there has not been made much research on the permeability of the recycled concrete and not even after the heating and cooling process at high temperature.

1.4.2. The influence of the recycled aggregates on the mechanical behavior of concrete subjected to high temperature

At ambient temperature, the concrete is considered a fragile material. Its behaviour is asymmetric in tension and compression in the sense that its tensile strength is generally negligible compressive. The uniaxial compression test is used to observe a softening character of the material once passed the peak with anisotropic behaviour (high expansion in the direction perpendicular to the load). Subjected to high temperature, the concrete undergoes irreversible damage. Under the effect of different physico-chemical changes that take place within the material during the temperature rise, the mechanical performance of concrete decreases.

This chapter focuses on the impact on the compressive strength and tensile strength and the modulus of elasticity of the normally heated concrete. That is because there has not been done previous research on concrete with recycled aggregates subjected to high temperatures.

1.4.2.1. Evolution of the compressive strength

The compressive strength is one of the most important properties to characterize the concrete. It generally decrease with increasing temperature.

The mechanical tests can be operated hot or after cooling the concrete. Usually the hot tests allow to assess the current behaviour of the fire. Those made after cooling indicate “post-fire” values, useful for assessing the residual capacity of a structure and repair options. The values of residual compressive strength are generally lower than those which are measured in the hot proceedings (Figure 1.8). The cooling induces additional
Thermal loading and the hydration of CaO. It is accompanied by the humidity of the ambient air and an appreciable volume increase (accompanied by cracking).

![Figure 1.8: Evolution of the relative compressive strength of concrete based on the type of test performed [Hage 2004].](image)

The heating rate plays an important role on the residual compressive strength of concrete. According to studies by Khoury [Khou 2000], he found that applying a slow speed of heating allows a more complete chemical transformation in concrete and therefore a significant reduction in resistance. Whether hot or after cooling, the evolution of the compressive strength can be divided into three phases. The first phase, from ambient temperature to 100-200°C (according to the authors) is marked by a decrease of 20 to 30% of the resistance [Hage 2004]. The second phase 100-250°C or 200-350°C (according to the authors), is characterized by an improvement or maintenance of resistance.

The evolution of the compressive strength as a function of temperature can be explained by the following phenomena. During the first phase, the expansion of water causes a separation of sheets of CSH. This separation generates a reduction in cohesive forces of van der Waals. This surface energy reduction favors the formation of silanol groups (Si-OH) which have weak bonding forces. The weakening of the bonds between hydrates decreases the shear strength between the grains which cause a reduction in the compressive strength [Xing 2011]. During the second phase, the removal of water can
cause a re-increase bond strengths between hydrates with the disappearance of silanol groups. The increase in surface energy is the cause of the increase of the compressive strength [Dias 1990]. During the third phase, the dehydroxylation of portlandite and especially the differential thermal expansion concrete-paste-aggregate are causing cracks, firstly concrete-paste/aggregates and propagating in the pulp interface, consequent decrease of almost linear compression resistance with temperature.

The only study of recycled concrete was carried out by Huang [Huan 2006], Xiao and Huang [Xi H 2006]. They evaluated the influence of the recycled concrete aggregates on the fire resistance and residual compressive strength of concrete. Five groups of concrete with different recycled aggregates replacement ratios, i.e., 0, 30, 70 and 100% were exposed to temperatures from 20°C to 800°C. The conclusions (according to the authors) were, that the recycled concrete has a good resistance against explosive spalling and that the recycled concrete has some influence on the variation of the residual compressive strength subjected to different temperatures, shown in Figure 19. Prior to 300°C, the residual compressive strengths for all concretes follow a similar trend: at this stage the recycled concrete aggregates has only a slight influence. However, after 300°C, the influence of recycled concrete aggregates begin to be more remarkable: for concrete with 30% recycled concrete aggregates, similar to the conventional concrete, the residual compressive strength decreases sharply; while for concrete with 50%, 70% and 100% recycled concrete aggregates, the residual compressive strength tends to increase until 500°C and then begins to decrease [Xiao 2006].
1.4.2.2. Evolution of the tensile strength of concrete

There are few results concerning the evolution of the tensile strength as a function of temperature. This can be explained by the macroscopic behavior of structures and the experimental difficulties caused by the direct tensile test (hot alignment problems of the specimen bending parasite influenced bearings, rigidity of the press, thermal gradients, etc.). However, the scale of the material, knowledge of the tensile strength of concrete is needed to determine the damage and crack opening. For practical reasons of experimental simplicity, most researchers suggest the results of changes in the tensile strength of concrete from splitting tests or bending.

Hager [Hage 2004] conducted tests of direct tension during the thermal cycle at the end of the stabilization period of the temperature plateau on BHP (100 MPa). It was observed that the obtained tensile strength rose with hot temperature increase up to 400°C (Figure 1.10 a).
Conversely, Gambarova (2003) found that the tensile strength of measured concrete heat decreases with rising temperature (Figure 1.10 b) [Mind 2009].

Studies on the residual tensile agree on the strength reduction with increasing temperature. This decrease is almost linear, and is greater than the compressive strength [Noum 1995] [Li 2004] [Kane 2007] [Chan 2006]. Thelandersson (1971) noted that a more compact concrete has a lower decrease in tensile strength [Schn 1985]. What does not really dependent on the heating rate.

Even here no studies were found regarding the tensile tests on specimens of recycled concrete subjected to high temperatures. Were found, however, tests of recycled concrete at room temperature.

Liu et al. [Liu 2005], Xiao and Lan [Xi L 2006] evaluated the tensile behaviour of concrete with different recycled concrete aggregates contents through a direct tensile test. Their results shows the relative tensile strength, which are defined as the ratio of the uniaxial tensile strength of the recycled concrete to that of the control concrete, are presented in Figure 1.11. This figure shows that, as the recycled concrete aggregates increases, the uniaxial tensile strength decreases.

Concrete consisting of 100% of recycled concrete aggregates, the tensile strengths can only reach 88% and 69% of the control concrete. They have observed that the influence of recycled concrete aggregates can be neglected when it does not exceed 20%.
The results of the tensile strength of recycled concrete are very few. Another very important aspect to examine is the quality of the paste/aggregate interface (critical in the mechanism of tensile failure).

1.4.2.3. The evolution of the modulus of elasticity of the concrete

The elastic modulus reflects the rigidity of the concrete. This is severely altered by the temperature rise. Is there a loss of evaporable water below 100°C, then the concrete tends to increase the compressive strength and decreases the modulus of elasticity. Heat affects similarly the elasticity and strength of concrete above 100°C module. Many experimental studies have shown a gradual decrease in the modulus of elasticity in compression with temperature [Noum 1995] [Kane 2007]. [Noum 1995] noted that the evolution curve of the modulus of elasticity of the BHP is very close and seems similar to that of ordinary concrete. Hager [Hage 2004] noted that the E / C had no significant influence on the evolution of the elastic modulus up to 600°C with a heating rate of 1°C / min.
No studies were found that analyze the evolution of elastic modulus for recycled concrete subjected to high temperatures, but only those tested at room temperature.

Xiao [Xiao 2005] evaluated the elastic modulus of concrete with different replacement percentage \( r \) of recycled aggregates. Figure 1.12 shows that the elastic modulus of the recycled concrete aggregates is lower than that of the normal concrete (i.e., \( r = 0\% \)), and it decreases with an increased recycled concrete aggregates replacement percentage. When the recycled aggregates replacement percentage is 100\%, the author has obtained a elastic modulus reduced by 45\%. This is the consequence of the application of the recycled aggregates with a lower elastic modulus than that of the natural coarse aggregates.

![Figure 1.12: Elastic modulus of recycled concrete [Xiao 2005].](image)

In previous studies, Topcu [Top 1995] found that the reduction of the elastic modulus of recycled concrete aggregates was 80\%, while Frondistou and Hansen [Etxe 2007] reported that the reductions of the elastic modulus of recycled concrete aggregates were 33\% and 14–28\%. The main reason for the differences in the reduction of the elastic modulus depends on the different elastic modulus of recycled concrete aggregates used by the investigators.
CHAPTER 2 - EXPERIMENTAL METHODOLOGY

2.1. THE CHARACTERISTICS OF THE USED MATERIALS

We made one categorie of concrete of the mixture of cement, water, superplasticizer and two types of aggregates: a limestone sand used as fine aggregate and the 100% of recycled aggregates as coarse aggregates. One formulation was made with a W / C ratio of 0.3. The characteristics of the various components are described below:

2.1.1. Cement

It comes from the plant to Calcia Villiers Bouin owned Italcementi group. This cement is referenced CEM I 52.5 N CE CP2 NF. Its true class 61.3 MPa, the absolute density of 3130 kg/m$^3$ and surface Blaine is 3590 cm$^2$/g. It is composed of 66.9% C$_3$S, 10.7% C$_2$S, C$_3$A 8.4% and 7.6% of C$_4$AF. Its chemical composition is shown in Table 2.1.

| Elemental composition | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | TiO$_2$ | MnO | CaO | MgO | SO$_3$ | K$_2$O | Na$_2$O | F$_2$O$_5$
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>20.23</td>
<td>4.29</td>
<td>2.35</td>
<td>0.25</td>
<td>0.02</td>
<td>63.67</td>
<td>3.88</td>
<td>2.80</td>
<td>0.69</td>
<td>0.14</td>
<td>0.31</td>
</tr>
</tbody>
</table>

Table 2.1: Chemical composition of cement.

Dosage of cement

According to the norm NF EN 197-1 the minimum cement content must meet the following requirements:
• durability;
• protection of armor;
• workability.

So the minimum quantity is expressed by the formula:

\[ C_{\text{min}} = \frac{250 + 10 \times \sigma_{C28d} [\text{MPa}]}{\sqrt{D_{\text{max}} [\text{mm}]}} \]

The proportion of cement is calculated according to the desired workability. See the following graph (Figure 2.1):

*Figure 2.1: Graphic used to evaluate the quantity of cement based on the C/W ratio and the desired workability (slump of Abrams cone).*
2.1.2. Water

The used water is that of the distribution of drinking water to the urban community of Cergy-Pontoise. Its density is 1000 kg/m$^3$.

**Dosage of water**

To obtain an approximate dosage of the amount of water using the graph according to the desired workability (Figure 2.1) and the Bolowey formula shown below:

$$\sigma_{C28d} = G \times \sigma_C \times \left( \frac{C}{W} - 0.5 \right)$$

Where $\sigma_{C28d}$ is the average compressive strength of concrete at 28 days [MPa], $\sigma_C$ the true class cement 28 days [MPa], $C$ is the dosage of cement of concrete [Kg/m$^3$], $E$ is the dosage of water of concrete [Kg/m$^3$] and $G$ is the coefficient granular (Table 2.2) depending on the quality and the maximum aggregate size.

<table>
<thead>
<tr>
<th>Quality aggregates</th>
<th>Dimension D of aggregates</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fins (D &lt;16 mm)</td>
</tr>
<tr>
<td>Excellent</td>
<td>0,55</td>
</tr>
<tr>
<td>Good common</td>
<td>0,45</td>
</tr>
<tr>
<td>Fair</td>
<td>0,35</td>
</tr>
</tbody>
</table>

*Table 2.2: Coefficient granular G depending on the quality and the size of aggregate D.*

*These values assume that the compaction of the concrete will be done in good conditions (vibration in principle)*

After obtaining the approximate quantity of water, we proceed with the corrections:

- correction of the dosage of water in function of the diameter maximum of the aggregates «D$_{max}$» (Table 2.3).
Table 2.3: Correction on the percentage water content in accordance with the maximum aggregates dimension $D_{\text{max}}$ (if $D_{\text{max}} \neq 25\text{mm}$).

Thus obtaining the value correct:

$$E_{\text{correct}} = E \times \left(1 + \frac{C_r}{100}\right)$$

- Correction of water due to the presence of absorbed water in the aggregates (Table 2.4).

Table 2.4: Approximate water content of the aggregates contained in a $m^3$ of material in liters.

<table>
<thead>
<tr>
<th>Apparent degree of humidity</th>
<th>Adsorbed water in $1m^3$ of material in liters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry</td>
<td>0-20</td>
</tr>
<tr>
<td>Wet</td>
<td>40-60</td>
</tr>
<tr>
<td>Very wet</td>
<td>80-100</td>
</tr>
<tr>
<td>Saturated</td>
<td>120-140</td>
</tr>
</tbody>
</table>

At the end we obtained the correct quantity of water to be added:

$$E_{\text{total}}[l/m^3] = E_{\text{correct}} - E_{\text{adsorbed}}$$

2.1.3. Superplasticizer

The used additive in the manufacture of our concrete to ensure the adequate flow of implementation is the superplasticizer © CIMFLUID 3002 (TM © SIKA AXIM
Italcimenti group) which belongs to the family of superplasticizers / high water reducers. It is incorporated in the mixed water. This new generation of additive, which bases on modified polycarboxylate, is a beige opaque liquid. The characteristics of this superplasticizer are given in Table 2.5. Its dosage varies from 0.2 to 2.5 kg per 100 kg of cement according to the desired effect.

<table>
<thead>
<tr>
<th>Volumic mass</th>
<th>PH</th>
<th>Sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,070 kg/dm³ ± 0.020 (20°C)</td>
<td>5.5 ± 1.0</td>
<td>30.0 % ± 1.5 %</td>
</tr>
</tbody>
</table>

*Table 2.5: Specifications of high water reducing superplasticizer © CIMFLUID 3002.*

2.1.4. **Aggregates**

The recycled aggregates which are used for the production of recycled concrete is guided by previous studies. The work of the PhD Zhi Xing [Xing 2011] was used as a comparative model, which estimated the influence of the mineralogical nature of the aggregates on the behavior of concrete at high temperatures. Between the three types of aggregates that he have been used, were chosen those of silico-carcareous nature, the most widespread and those with better performance both in mechanical tests and at both high temperatures. For the production of concrete, from which were obtained the coarse aggregates, were used aggregates classes generally accepted in the field of civil engineering. The recycled aggregates are composed of alluvial aggregate semi-crushed silico-calcareous (78 Yvelines, France) with a particle size range of 0/4 and 6.3/20 mm. Of the produced concrete with these aggregates we know the composition of concrete (Table 2.6) and all the mechanical, physical and thermal property (Table 2.7). After a curing period of 90 days at a temperature of 20 ± 2 °C, the cylindrical specimens were broken with a compression press until we obtained the desired diameter of the aggregate.
2.1.4.1. Granulometric characteristics

The physical and mechanical properties of concrete depend on several factors. The manufacturer's main achievement is to obtain resistant, waterproof and durable concrete. To achieve this, the curves of granulometric analysis are used to enable us to acquire the best compactness of mixed aggregates. The gradation of different aggregates were determined through representative samples of gravel, crushed stone and sand
Thermical and mechanical behavior of recycled concrete aggregates and subjected to high temperatures

according to standard NF EN 13285. The results of granulometric analysis of aggregates are shown in Figure 2.2.

The intersections of the grading curve and the reference line connect the ordinate 95% and 5% of successive grading curves give the density distribution of the various components of the aggregate. We determined the optimized granular composition containing 38% sand and 62% recycled aggregates coarse. These values were used for the formulation of our concrete.

2.1.4.2. The volumic mass and the coefficient of water absorption

The density of granules is a fundamental physical characteristic significantly influencing the mechanical properties of aggregates and therefore the performance of concrete. The coefficient of the water absorption of recycled aggregates is a very important factor, since these have a great power to absorb water. It influences the
amount of mixing water which is required to produce concrete. The actual densities and water absorption coefficients at 24 hours of aggregates used in this study are measured (Table 2.8) in the laboratory of mechanical and civil engineering materials (L2MGC) according to EN 1097-6.

<table>
<thead>
<tr>
<th>Type of aggregate</th>
<th>Sand [0/4mm]</th>
<th>Gravel [4/20mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volumic mass</td>
<td>2563</td>
<td>2340</td>
</tr>
<tr>
<td>(Kg/m$^3$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water absorption</td>
<td>1.05</td>
<td>7.01</td>
</tr>
<tr>
<td>coefficient (%)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Table 2.8: Class granular, volumic mass and water absorption coefficient of recycled aggregate studied.*
2.2. PREPARATION AND PACKAGING OF CONCRETE SAMPLES

2.2.1. The formulation and composition of concrete

The objective of this study is to analyze the influence of the recycled aggregates on the behavior of concrete subjected to high temperatures. We conducted one simple formulation using the recycled aggregates and limestone sand consisting of water/cement ratio equal to 0.3.

The dosage of this cement concrete is 500kg/m3. The composition of concrete is obtained by the method of formulation of Dreux Grorisse [Dreu 2002]. The additive amount was been determined by preliminary tests in order to obtain comparable workability for the concrete, performed by the PhD Zhi Xing [Xing 2011]. The subsidence of Abrams cone of studied concrete was chosen in S4 class (between 16 and 20 cm). We present the composition of the used concrete in Table 2.9.

<table>
<thead>
<tr>
<th>Material</th>
<th>Amount [Kg/m³]</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement</td>
<td>500,00</td>
<td></td>
</tr>
<tr>
<td>Gravel</td>
<td>1015,56</td>
<td></td>
</tr>
<tr>
<td>Sand</td>
<td>597,18</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>150,00</td>
<td></td>
</tr>
<tr>
<td>Raport W/C</td>
<td>0,30</td>
<td></td>
</tr>
<tr>
<td>Superplasticizer [kg/m³]</td>
<td>1,65</td>
<td>[kg/m³]</td>
</tr>
<tr>
<td>[% of SP/C]</td>
<td>0,33</td>
<td></td>
</tr>
<tr>
<td>Volumic Mass [Kg/m³]</td>
<td>2333</td>
<td></td>
</tr>
</tbody>
</table>

*Table 2.9: Composition of the concrete of the study (data for 1m³).*
To determine the evolution of the thermal properties of recycled concrete during heating, we made specimen of various sizes for the relevant tests listed below:

- 6 cylinders of dimensions 16x32 cm (3 with thermocouples);
- 5 cylinders of dimensions 15x30 cm;
- 53 cylinders of dimensions 11x22 cm.

All the specimen have the same volume proportion of components (cement, sand, water and superplasticizer). By analyzing the thermal properties of the made concrete, we can deduce the thermal properties of recycled aggregates during heating.

2.2.2. The manufacturing process of recycled concrete

The protocol steps of the concrete manufactured are the following:

- We put the right amount of recycled aggregates into water. But, 24 hours before made the concrete, so that the aggregates saturate.

- We then calculated the water content of the materials (sand and gravel) using an oven at a temperature of 105°C. This step allows the correct amount of water to be introduced into the mixer.

- We finally arranged the saturated aggregates in a strainer in order to obtain a “saturated/dry surface” aggregate (Figure 2.3).
• Pre-wetting of the mixer for limiting absorption of water by the walls thereof.

• Introduction of the components starting with recycled aggregates, sand and cement in the mixer Couvrot type rotary blade and a maximum capacity of 90 liters (Figure 2.4).

• Mixing of the aggregate and the binder without adding water for one minute.
• Mix the water with the superplasticizer in a container, then adding a little water the same time while mixing for some minutes.

• Measurement of the Abrams cone slump, then drain concrete mixer wheelbarrow. The standard concrete slump is limited to 2 cm from the target value to ensure similarity to each mixture.

• Pouring fresh concrete in the mold in two layers. Each layer is vibrated for 20 seconds. The specimens were finally protected from desiccation by a plastic cover, weighed and stored on wooden pallets at room temperature.

2.2.3. Storage condition

The storage conditions of the test are made with regard to the recommendation of RILEM TC-129. All samples were stored at a temperature of 20 ± 2°C during the first day after casting in molds without water exchange with the outside (Figure 2.5). They are then stored in plastic bags that maintain a humidity of 100% (Figure 2.6). The specimen aged 90 days.

Figure 2.5: Unpacking of cylinders.  
Figure 2.6: Storage in plastic bag.
2.3. THE METHODOLOGY AND THE EXPERIMENTAL PROGRAMM

2.3.1. Heating-cooling cycles

The concrete samples undergo cycles of a heating-cooling phase consisting of a rise in temperature, a stabilization phase at a constant temperature and a third stage temperature lowered to room temperature. The heating ramps use the same cooling rate of 1°C/min. This rate is often used in the literature for the dimensions of our specimens. We apply four cycles of heating and cooling from room temperature (20°C) to 150, 300, 450 and 750°C for concrete. Each heating plateau is used an maximum temperature for one hour. The set temperature corresponds to the removal of free water (150°C), at the end of the dehydration of CSH (300°C) at the beginning of decomposition of Portlandite (450°C) and the phenomenon of decarbonation (750°C). Figure 2.7 shows the changes of temperature versus time for the 4 studied heating cycles. The setpoint temperatures are indicated and the actual development of the surface temperature of the concrete in the heating cycles. We observe that the actual temperature is very close to the set temperature during the heating cycle except for the cooling phase where it is slower, where it limits the risk of a thermal shock.

![Figure 2.7: Cycles of heating and cooling imposed on recycled concrete.](image-url)
The cycles of heating/cooling are performed in a programmable oven dimensions 1.3x1.0x1.0 m (Figure 2.8 a), where the temperature can reach 1100°C. This oven is supervised by the EUROTHERM program controller which is connected to a thermocouple placed on the surface of a specimen. A fan associated with heating is used to regulate and homogenize the temperature of the air flow between the heaters. For the acquisition of the temperature data, a central automatic acquisition HP323 is used (Figure 2.8 b). It has 22 tracks to record continuously the temperature inside the oven and within samples. This logger is controlled by a computer. The type K thermocouples placed on different concrete specimen allows us to ensure the temperature uniformity in the oven (Figure 2.9).

Figure 2.8: a) The oven, left; b) acquisition device, right.

Figure 2.9: Arrangement of concrete specimens in the oven.
2.3.2. The characterization of the behavior of concrete subjected to high temperature

The objective of the experimental program was to characterize the behavior of the concrete according to the heating temperature. The studied properties are: the residual mechanical ones (compressive strength, elasticity and tensile modulus), loss of weight, porosity of water, the evolution of the aggregate / matrix interface of the concrete and the evolution of cracking. Table 2.10 summarizes the various tests and the number of samples used for measurement for each temperature (20°C, 150°C, 300°C, 450°C and 750°C). The specimens Ø 16x32 cm are intended for observation of cracking of the concrete for each cycle of heating and cooling. The specimen Ø 11x22 cm are reserved for the measuring of a compression test, for the splitting tensile and for the elastic modulus. The specimens Ø 15x30 are intended for the study of permeability. In addition, we have crafted some specimen dimension Ø 16x32 cm in the center, which we placed K-type thermocouples to measure the temperature evolution and to characterize the thermal gradients of the concrete during the heat treatment.

Table 2.10: Overview of tests for recycled concrete.

<table>
<thead>
<tr>
<th>Tests</th>
<th>Dimension</th>
<th>Temperature</th>
<th>Measuring device</th>
<th>Quantity of test</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compression resistance</td>
<td>Ø 11x22 cm</td>
<td>For each temperature 20, 150, 300, 450 and 750°C</td>
<td>Hydraulic press</td>
<td>3</td>
</tr>
<tr>
<td>Direct tensile resistance</td>
<td>Ø 11x22 cm</td>
<td></td>
<td>Pundit lab</td>
<td>3</td>
</tr>
<tr>
<td>Elastic modulus</td>
<td>Half of Ø 11x22 cm</td>
<td></td>
<td>Electronic microscope</td>
<td>3</td>
</tr>
<tr>
<td>Evolution of fissuration</td>
<td>Ø 16x5 cm</td>
<td></td>
<td>Desiccator, vacuum pump and balance</td>
<td>1</td>
</tr>
<tr>
<td>Permeability test</td>
<td>Ø 15x30 cm</td>
<td></td>
<td>Acquisition center with thermocouples</td>
<td>3</td>
</tr>
<tr>
<td>Temperature difference of surface/center of the specimen</td>
<td>Ø 16x32 cm</td>
<td>750°C</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.4. THE EXPERIMENTAL PROCEDURE
2.4.1. Measurements of the mechanical properties of recycled concrete subjected to high temperatures

At the age of 90 days, the specimen of recycled concrete were subjected to heating/cooling cycles in the oven and were cooled before performing mechanical tests such as uniaxial compressive and tensile splitting. The evolution of the residual mechanical properties of concrete were analyzed. These tests were all done on a hydraulic press INSTRON 3000 kN assisted by a computer (Figure 2.10) to L2MGC.

![Figure 2.10: The hydraulic press for the mechanical test.](image)

2.4.1.1. The uniaxial compression tests

After each heat treatment cycle for each temperature three specimens of Ø11x22 cm are surfaced with a sulfur based material of high performance. Once the base of samples contain sulfur, non had to wait at least 2 hours to complete the mechanical test. It's necessary to delay the hardening of sulfur. Since the RILEM recommendation states that testing must be performed within two hours following the release of specimens from the oven to prevent rehydration, the samples are re-packed in sealed plastic bags to minimize the water exchange between the specimen and the ambient air.
One of the three cylinder specimens Ø 11x22 is placed on the press and mechanically charged at a rate of 0.5 MPa/s to obtain the breaking strength under compression according to EN 12390-3. Three trials were conducted for recycled concrete for each set temperature.

2.4.1.2. The splitting tensile tests

We chose the splitting tensile test, also called Brazilian test, for the shape of specimen to determine the tensile strength of concrete (Figure 2.11).

The preparation and storage of specimens for the tensile test in the oven, are the same as the ones mentioned above. The norm EN 12390-6 is respected to characterize the splitting tensile strength. We use two hardboard strips on two surfaces in contact with the specimen, because the applied compressive force to the specimen will then better be distributed. This test is performed on three cylindrical specimen Ø 11x22 cm for each cycle of heating and cooling. The formula that allows to obtain the tensile stress is the following:

\[ f_t = \frac{2 \cdot P}{\pi \cdot \varnothing \cdot L} \]

\( P \) is the applied maximum force of the press on the measuring cylinder load. \( \varnothing \) is the diameter of the cylinder and \( L \) represents the length of the cylinder.

Figure 2.11: Schematic of the splitting tensile test.
2.4.1.3. **The measurement of Young's modulus**

The preparation and storage of specimens for the measurement of Young's modulus in the oven, are the same as the ones mentioned above. The respected norm ISO 6721-1 is follows the dynamic method. The dynamic method is to use the Pundit Lab (Figure 2.12), is an ultrasonic pulse velocity. This is a device to measure the elastic properties or strength of concrete. For this test we utilized four half specimens of Ø 11x22 to have a better reliability.

![Image of Pundit Lab](image.png)

*Figure 2.12: The instrument Pundit Lab for measure the elastic modulus.*

2.4.2. **The measurements of the physical and thermal properties of concrete subjected to high temperatures**

2.4.2.1. **The measurement of the weight loss of the concrete**

The measurement of mass loss is an important indicator, especially for the recycled concrete, of the damage sustained by the material as a function of the heat treatment. It allows to determine the evolution of the weight of the recycled concrete by the difference between the weight before and after each heating and cooling cycle.

The mass after heating is measured immediately after the cooling in order to avoid the rehydration during the storage in ambient air. This measured mass loss at the end of the heating-cooling cycle helps to quantify the lost material during thermal cycling.
Loss in mass percentage mass is obtained as follows:

\[ m_i = \frac{m_{ai} - m_{Ti}}{m_{ai}} \times 100\% \]

With \( m_i \) represents the mass loss in %, \( m_{ai} \) the mass of the sample at room temperature before heating and \( m_{Ti} \) the mass of the cooled after the heating-cooling cycle sample \( Ti \).

The concrete specimens are weighed at the end of the casting, after being removed from the mold, before and after the heat treatment in the oven. While heating some specimen were damaged by scaling phenomena and/or burst. Only specimen without apparent damage are used to take stock of the loss of mass.

The mass loss is measured on all cylindrical specimen with a balance. Following these measures, interesting information on the quantity of water are actually lost by the recycled concrete during heating and cooling are obtained.

### 2.4.2.2. The measurement of the porosity and the density of the concrete

Measuring the porosity allows to assess the degradation of the microstructure of the material caused by the temperature. It quantifies the volume of open pores without the determination of the particle size distribution of the pores.

The concrete specimens have a size of \( \frac{1}{4} \) of a cylinder of diameter 16 cm and a height 5 cm pre-cut (Figure 2.13). At least three samples were measured for each heating-cooling cycle.
Figure 2.13: A ¼ of a cylinder of Ø 16x5.

The samples are kept warm first in waterproof bags and then in an oven at a temperature of 80°C at a constant mass before the measurement. The test is executed dry when the difference between two successive measurements of mass spaced 24 hours is about the accuracy of the used scale (0.01g for the concrete). When samples are completely dry, they are immersed into water. To have a good saturation of the sample, the samples are placed in a sealed glass bell imposed to a pressure of 90 mbar while using a vacuum pump (Figure 2.14). The sample is then kept overnight (about 15 hours) in this state. Afterwards the water is introduced gradually until the sample is immersed in about 20 mm in height. This operation is designed to trap the minimum air in the pores of the granules during the immersion of the sample. The pressure in the bell is maintained for 24 hours. During this period of time the water is supposed to fill the entire pore network of the concrete. Following this period, the sample is considered saturated and weighed in water with a hydrostatic balance.
The determination of the porosity and bulk density of the recycled concrete is carried out by using the following equations:

\[
\varphi_{\text{concrete}} = \frac{m_{\text{sat-concrete}} - m_{\text{dry-concrete}}}{m_{\text{sat-concrete}} - m_{\text{imm-concrete}}}
\]

\[
\rho_{\text{app-concrete}} = \frac{m_{\text{dry-concrete}}}{m_{\text{sat-concrete}} - m_{\text{imm-concrete}}}
\]

Where \(m_{\text{sat-concrete}}\) and \(m_{\text{imm-concrete}}\) are a saturated specimen measured in air and mass in water, \(m_{\text{dry-concrete}}\) is the dry weight after drying in an oven at the temperature 80 °C.

Porosities are measured for all cycles, but the sample used at 750°C is too friable so, it does not achieve the sufficiently accurate measurements.

2.4.2.3. The measurement of the permeability of the concrete

A concrete structure is considered to be of adequate durability, if it performs according to its intended level of functionality and serviceability over an expected or predicted life cycle. Durable concrete must have the ability to withstand the potentially deteriorative conditions to which it can reasonably be expected to be exposed. In terms of
deterioration of concrete (due to physical or chemical causes), the mobility of fluids or gases through the concrete are nearly always involved. The overall susceptibility, or penetrability of a concrete structure, especially when compounded by additional environmental or exposure challenges, is the key to its ultimate serviceability and durability.

The overall permeability of concrete to water is a function of the permeability of the paste, the permeability and gradation of the aggregate, and the relative proportion of paste to aggregate. Low porosity / permeability / penetrability of concrete to moisture and gas is the first line of defense against: frost damage, acid attack, sulfate attack, corrosion of steel embedment and reinforcements, carbonation, alkali-aggregate reaction, and efflorescence to name a few of the most prominent concrete ailments. Here the permeability of the paste is important because the paste envelopes all constituents in the concrete. Paste permeability is related to water/cement (w/c) ratio and the degree of cement hydration or length of moist-curing. A low-permeability concrete requires a low w/c ratio and adequate moist-curing.

The measure of the concrete permeability falls in to three categories: hydraulic permeability which is the movement of water through concrete. Gas permeability which is the movement of air through concrete. The third involves the movement of electric charge (chloride-ion permeability).

The norm XP P18-463 indicates the use of the gas method for measuring the permeability.

This test method involves the establishment of a steady-state flow condition in a cylindrical concrete specimen of dimension Ø 15x5cm housed in a tri-axial permeability cell. A pressure gradient is maintained across the sample with one end exposed to ambient pressure and the opposite end at the test drive pressure, as shown in Figure 2.15. A radial confining pressure is maintained around the specimen.
Calculating the permeability of each test specimen by the formula:

\[ K_x = \frac{2 \cdot P_0 \cdot Q \cdot L \cdot \mu}{A(P_0^2 - P_1^2)} \]

\( Q \) is the gas flow rate, \( P_0 \) is the absolute pressure at the inlet, \( P_1 \) is the absolute pressure at the exit (atmospheric pressure), \( \mu \) is the dynamic viscosity of the gas used at the test temperature, \( L \) is the thickness of the specimen and \( A \) is the area of specimen.

2.4.3. The measurement of the thermal properties of concrete

2.4.3.1. The thermal gradient between the surface and the core of the recycled concrete specimen

Two thermocouples (type K.SV/SV.2 × 0.07mm²) were used for the test (Figure 2.16 a) to determine changes in the temperature gradient in the concrete during heating cycles. A thermocouple is embedded in the center of the test piece during the casting of the concrete. The second is placed on the surface of the test piece before the heating cycle (Figure 2.16 b). The temperature difference between the surface and the core of the specimen and the evolution of the temperature gradient of recycled concretes are monitored.
The temperature is measured at regular intervals during the heating-cooling cycle on cylindrical specimens of size Ø 16x32 cm and is recorded by the computer which acts as a central data acquisition (Figure 2.16 b).

Figure 2.16: Concrete specimen with thermocouples: a) position of the thermocouples in a concrete specimen; b) position of a thermocouple on the surface of concrete.
CHAPTER 3 - BEHAVIOR OF RECYCLED CONCRETE AT HIGH TEMPERATURES

This chapter focuses on the study of the influence of the recycled aggregates on the behavior of concrete subjected to different cycles of heating and cooling presented in Chapter 2. A type of recycled aggregate was used of silico-calcareous nature, the water/cement ratio (W/C) is 0.3. The obtained recycled concrete was subjected to thermal cycles of different amplitudes which allow us to distinguish the influence of the recycled aggregates according to the quality of the cement matrix on the evolution of the different properties with temperature. The cracking of the interface between old paste/aggregate and new/old paste after different thermal loads is investigated at microscopic level.

After different thermal cycles, we studied the behavior of concrete under mechanical stress. Compression tests were conducted, tractions where we measure the tensile strength and tests for the calculation of the modulus of elasticity. Furthermore the influence of thermal stresses on the mechanical damage of concrete is analyzed, given the observations of the cracking of concrete. We then present the evolution of porosity and mass loss of concrete depending on the heating temperature. These characteristics are important to compare the state of degradation of concrete depending on the recycled aggregates and the quality of the various interfaces. The thermal properties of concrete are strongly influenced by those recycled aggregates. We study the differences induced on the thermal conductivity and specific heat by the nature of the aggregates at room temperature and during heating. The thermal properties of concrete govern the temperature variations in the sample and therefore the intensity of the stresses due to the thermal gradient. We are also interested in the evolution of the thermal conductivity after cooling, which is an indicator of damage to the microstructure.
3.1. COMPARISON OF DEGRADATION OF RECYCLED CONCRETE WITH NORMAL CONCRETE

3.1.1 Evolution of degradation of recycled concrete specimens according to the heating temperature

The photos shown in Figure 3.1 shows the state after the test cycles heating/cooling. The first cracks are observed with the naked eye after the heating-cooling cycle of 300°C. Chipping of the surface (often near large aggregates) are observed on recycled concrete after the heating cycle of 750 °C (Figure 3.1).

Explosive bursts are not observed on any specimens and with whatever dimensions of recycled concrete, perhaps because the rate of heating/cooling of each cycle was slow enough to avoid explosions.

Given that recycled aggregates were composed of silico-calcareous aggregates, the concrete specimens heated to 750°C are crumbled in part several days after the cooling following the hydration of CaO to Ca(OH)$_2$ in the mortar and aggregates. The formation of portlandite is associated with an increase of volume and results in the disintegration of the specimen.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Specimens Ø16x32cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>150°C</td>
<td>![Specimen image]</td>
</tr>
<tr>
<td>Temperature</td>
<td>Specimens Ø16x32cm</td>
</tr>
<tr>
<td>-------------</td>
<td>-------------------</td>
</tr>
<tr>
<td>300°C</td>
<td><img src="image1" alt="Specimens Ø16x32cm after 300°C" /></td>
</tr>
<tr>
<td>450°C</td>
<td><img src="image2" alt="Specimens Ø16x32cm after 450°C" /></td>
</tr>
<tr>
<td>750°C</td>
<td><img src="image3" alt="Specimens Ø16x32cm after 750°C" /></td>
</tr>
</tbody>
</table>

*Figure 3.1: Specimens Ø16x32cm after heating cycles.*
3.1.2 Evolution of cracking at interfaces new paste/old paste/granulates according to the heating temperature

After the heating/cooling cycles of 300°C, 450°C and 750°C, we observed the evolution of the damage of the surface of a slice of concrete with dimensions Ø16x5cm previously cut before heating. The state of aggregates and their interfaces with the dough is observed firstly on the entire sample to the naked eye and at a later time with a binocular microscope.

After the heating cycle to 300°C, the cracks are hard to see with the naked eye (Figure 3.2), but it’s possible to see small cracks with a standard lens. The silica aggregates were brown, now their colour is reddish. The observation is needed under a binocular microscope.

After the heating cycle to 450°C with the naked eye, we note many cracks in the cement matrix (Figure 3.3). The limestone aggregates are intact, while the interface appears a bit cracked. The vast majority of silica aggregates are cracked in several directions. Wide cracks are visible in the mortar (radial cracks) and interface (tangential cracks), but not in the interfacial zone between new paste and old paste of the recycled aggregates. As we can see, the cracks passes through the recycled aggregate and it doesn’t turn around like natural aggregates.

After the heating to 750°C, we observe with the naked eye a stronger concrete deterioration with an increase of the cracks opening (Figure 3.4). The limestone aggregates are white and cracked. Instead the silica aggregates colour is red. The cracking is more pronounced than BRM 0,3 at 450°C (Figure 3.3). The cracks appear in the limestone aggregates and cracks are more numerous in the matrix. The previous cracks are enlarged and branched. There's no differentiation between new/old paste, the cracks cross the recycled aggregates as easy as the new mortar. At this temperature we find only the interface between the cement matrix and aggregates.
Figure 3.1: Degradation of the BRM 0.3 after heating to 150 °C. The cracks are not visible to the naked eye.
Figure 3.2: Degradation of the BRM 0.3 after heating to 300 °C. The cracks are not visible to the naked eye.
Figure 3.3: Degradation of the BRM 0.3 after heating to 450 °C. The cracks is visible to the naked eye.
Figure 3.4: Degradation of the BRM 0.3 after heating to 750 °C. The cracks is visible to the naked eye.
3.2. EVOLUTION OF MECHANICAL PROPERTIES OF RECYCLED CONCRETE

The mechanical properties such as compressive strength, tensile strength and modulus are determined according to Chapter 2 described before and after thermal stress procedures. These tests are used to analyze the influence of recycled aggregates on the residual mechanical behavior.

For a comparison we used the results of two other types of recycled concrete and a type of ordinary concrete. The concrete under review was called BRM 0,3 (Beton Recyclé Maison in French translated “recycled concrete house”) with a ratio W/C = 0,3. While other concretes used for a comparison are:

- BRC 0,3 concrete produced from PhD Cléo, using as coarse aggregates those from a demolished building. Therefore with an old concrete that we don’t know any characteristics and properties. The ratio W/C was 0.3 as that one in question.
- BRM 0,6 concrete produced from PhD Cléo, using as coarse aggregate with those used in question, but the ratio W/C = 0,6.
- BSC 0,3 ordinary concrete produced from PhD Zhi with silico-calcareous aggregates. This is the concrete from which the aggregates have been obtained to produce concrete under examination. The W/C ratio was the same, so 0,3.

3.2.1. Residual compressive strength

Table 3.1 shows the residual compressive strength obtained at 20°C after different heat treatments for recycled concrete (BRM 0,3). The compressive strength of recycled concrete at 90 days is the initial resistance of reference. It allows to calculate the residual relative resistance with \( \frac{f_{ct}(T)}{f_{ct}(20)} \).
Thermal and mechanical behavior of recycled concrete aggregates and subjected to high temperatures

Table 3.1: Residual compressive strength of recycled concrete.

<table>
<thead>
<tr>
<th>Compression</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>resistance</td>
<td>20°C</td>
</tr>
<tr>
<td>$f_{c(T)}$ [Mpa]</td>
<td>49,05</td>
</tr>
<tr>
<td>$f_{c(T)}/f_{c(20)}$</td>
<td>1</td>
</tr>
</tbody>
</table>

At room temperature, the resistance is around 49 MPa for recycled concrete. Figures 3.5 shows the variation of relative and absolute residual strengths depending on the heating temperature compared with other types of recycled concrete (BRC 0,3 and BRM 0,6) and the ordinary concrete (BSC 0,3).
In these figures, we can notice the two different areas of evolution which depend on the temperature of heating. One maintains a compressive strength up to 300°C and the other one decreases with increasing temperature. We can notice the same behavior for ordinary concrete subjected to high temperatures [Noum 1995] [Xing 2011].

For recycled concrete (BRM 0,3 in Figure 3.5), the evolution of the residual compressive strength increases up to 300°C. This behavior is very interesting. It can be explained by the fact that the hydration of the cement is very long and during the heating comes the elimination of free water. This water goes to hydrate the CSH of cement paste and this increases the resistance.

At 450°C the resistance of the recycled concrete is no more equal to 50% of its original strength, while at 750°C the compression strength drops to 15%.

Figure 3.5: Residual compressive strength of ordinary concrete depending on the heating temperature: a) absolute value, to the left; b) value relative to the right.
3.2.2. Residual tensile strength

Although concrete is not normally designed to resist efforts to direct tension, knowledge of the tensile strength allows to estimate the load that will cause cracking.

The evolution of tensile strength after different thermal loads enable to the estimation of the damage in the paste-aggregate interface depending on the heating temperature.

*Table 3.3* shows the residual tensile strengths obtained by tensile strength test of recycled concrete.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>20°C</th>
<th>150°C</th>
<th>300°C</th>
<th>450°C</th>
<th>750°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$f_{t(T)}$</td>
<td>5.30</td>
<td>4.69</td>
<td>3.94</td>
<td>2.06</td>
<td>0.55</td>
</tr>
<tr>
<td>$f_{t(T)}/f_{t(20)}$</td>
<td>1.00</td>
<td>0.88</td>
<td>0.74</td>
<td>0.39</td>
<td>0.10</td>
</tr>
</tbody>
</table>

*Table 3.3: Residual tensile strength by splitting studied of recycled concrete.*

**Tensile strength resistance**
Figure 3.6: Residual tensile strength by splitting of recycled concrete heated after thermal cycling: a) absolute value, left; b) relative value, right.

Figures 3.6 shows the variation of the residual tensile strength based on the temperature of recycled concrete. The tensile strengths of recycled concrete tested continues to decrease with increasing temperature.

If we compare the behavior of ordinary concrete with the behavior of the recycled concrete so we can notice which the residual tensile behavior of ordinary concrete is significantly better than the recycled concrete.

It’s important to notice the test performed at room temperature. The tensile strength of the recycled concrete (BRM 0,3) is higher than the ordinary concrete; even if the best are the aggregates obtained from the demolition of buildings that have a cement mortar even older. The adhesion between the old cement paste and the new one is better, this happens because they have the same composition and therefore they are compatible.

After the heating to 750°C, the residual tensile strength of the recycled concrete is only 10% compared to the initial resistance at 20°C. The loss of tensile strength of the recycled concrete is higher than the loss of compressive strength. This can be explained by the cracking of the paste-aggregate interface that has been highlighted in paragraph 2 in the study of cracking.
3.2.3. Residual elastic modulus

*Table 3.4* presents the values of the residual elastic modulus obtained at 20°C and after several cycles of thermal stress. These express the modulus of elasticity changes in rigidity of the material depending on the heating temperature.

<table>
<thead>
<tr>
<th>Elastic modulus</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>20°C</td>
</tr>
<tr>
<td>$E(T)$ [Gpa]</td>
<td>45,67</td>
</tr>
<tr>
<td>$E(T)/E(20)$</td>
<td>1</td>
</tr>
</tbody>
</table>

*Table 3.4: Residual elastic modulus of recycled concrete.*

---

**Elastic modulus**

![Diagram showing the change in elastic modulus with temperature for different materials.](image)
Thermical and mechanical behavior of recycled concrete aggregates and subjected to high temperatures

Figure 3.7: Residual modulus of elasticity of recycled concrete heated according to different temperature cycles: a) absolute value, to the left; b) value relative to the right. The elastic modulus decreases steadily with increasing heating temperature.

In Figure 3.7 can be observed the evolution of elastic modulus of recycled concrete after different thermal cycles. The elastic modulus decreases steadily with increasing heating temperature. After the heating at 750°C the recycled concrete has residual modulus at 4% of his initial module.

Thermal damage of the concrete is related to physico-chemical changes of the dough, but also depends heavily on the recycled aggregates. This study showed that after 300°C the recycled aggregate is critical to the loss of mechanical performance. The incompatibility of thermal deformation between the paste/aggregate and old/new pasta cause significant cracks that start from the interface aggregate/old pasta and old/new pasta which propagate in the cement matrix. The quality of the interface plays an important role in the resistance of concrete to thermal stress like as also the influence of the W/C ratio on the residual behavior of concrete.

In any case, the trend of elastic modulus of the recycled concrete in consideration (BRM 0.3) is almost the same as the ordinary concrete (BSC 0.3).

The mechanical tests are performed immediately after cooling. The residual resistance,
after the heating at 750°C, would have been lower if they had been tested several hours after leaving the oven. Indeed, hydration of CaO into Ca(OH)$_2$ is associated with an increase of volume and results in the disintegration of the specimen.
3.3. EVOLUTION OF THE PHYSICAL PROPERTIES OF CONCRETE

3.3.1. The mass loss of heated concrete

The exposure of concrete at high temperatures degrades the aggregate and cement paste. One of the consequences is the reduction in the density of concrete. The weight loss can include quantifying the free and bound water present before and after heating in a concrete specimen. The water is found bound in hydrates, adsorbed on the surface of the solid and free items in the pores of old and new paste. The free water has the property of being easily lost during thermal variations.

The value of weight loss was an average of measurements on every type of specimens for each temperature. The average value and the standard deviation of the mass loss of recycled concrete is listed in the following table (Table 3.5).

<table>
<thead>
<tr>
<th>Mass loss</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>20°C</td>
</tr>
<tr>
<td></td>
<td>0,00</td>
</tr>
</tbody>
</table>

*Table 3.5: Average mass loss of recycled concrete studied after heating.*

Despite of having used a low ratio W/C, the largest share of total water is contained in the recycled aggregates. From the diagram (Figure 3.8) we can see the highest loss of mass of recycled concrete. This can be partly explained by the large amount of water absorbed by the porous of recycled aggregate and the departure of bound water after 450°C. In fact, the recycled concrete is different from the ordinary concrete to an higher loss of free water up to 300 °C. After this temperature, the slope of the curves of the various concretes are almost similar. That’s why the bound water is always the same.
3.3.2. The porosity and mass density of recycled concrete

For lack of time this test was not performed, but it will be completed by PhD Cléo.

3.3.3. Permeability of recycled concrete

The results obtained had errors, due probably to the detection method. The test will be executed again from PhD Cléo.
3.4. EVOLUTION OF THE THERMAL PROPERTIES OF CONCRETE

The study of the thermal properties of concrete is essential to study the behavior of concrete at high temperature. We measure the temperature difference between the surface and the center of the specimens to analyze the distribution of heat within the recycled concrete during heating.

3.4.1 The temperature difference between the surface and the center of the recycled concrete specimen

During the preparation of concrete specimen, a thermocouple embedded in the center of cylindrical specimen dimensions Ø16x32cm. Another thermocouple is positioned on the surface of the specimen. The measurement of surface temperatures ($T_{\text{surface}}$) and the center of the specimen ($T_{\text{center}}$) has possible to determine the temperature difference ($T$) between the center and the surface of the test piece depending on the surface temperature.

The Figure 3.9 shows the temperature difference between the center and the surface of the specimen that increases to a peak at 335°C. Then it decreases up to 570°C and finally it increases again to another peak at 620°C. The temperature difference ($T$) is almost constant between the three specimen of the recycled concrete at 750°C. While regarding the source concrete, we have no data because the thermocouples were broken during the test.
The temperature range where there are the maximum temperature differences, corresponds to the inflection point of the curve of weight loss. From this temperature (335°C), the mass loss rate decreases because the most of the free water and bound water is gone. The heat consumption in a latent form, due to evaporation of water, delays the transfer of heat from the surface to the center of the specimen. The departure of most of the water between 100°C and 335°C by evaporation causes an increase of the temperature difference between the surface and the center, which reaches its maximum value at about 335°C. The temperature difference is lower for recycled concrete (105°C) than for the ordinary concrete (128°C) even if the recycled concrete contains more free water. Their higher porosity and permeability facilitate migration and drainage. Therefore, the peak of the ordinary concrete will be moved to the right in the graph due to the lower porosity of the aggregates and thus the higher difficulty in migration of free water.

It’s important to know that for the recycled concrete with a ratio W/C = 0,6 (BRM 0,6) the temperature difference between surface and the center is higher than the recycled
concrete in question with a ratio W/C = 0.3 (BRM 0.3). This happens because the amount of free ejected water is higher, then latent heat is higher. Naturally, the peak of BRM 0.6 also will be moved to the left of the graph (lower temperature) since for an higher W/C ratio corresponds a greater porosity.

The appearance of the second peak temperature difference at 620°C can be explained by the second stage of the decomposition of CSH gel and the formation of β-C₂S [Noum 1995].

Looking at the bottom of the chart (during cooling) can be seen a third peak. This happens at a temperature of 550°C because there is a rehydration of Portlandite.

\[
CaO + H_2O \rightarrow Ca(OH)_2 \text{ Portlandite}
\]
3.5 CONCLUSIONS

This chapter presents a comparative physical, thermal and mechanical analysis of concrete properties with recycled coarse aggregates exposed to cycles of heating and cooling at 150°C, 300°C, 450°C, 750°C with a heating rate of 0.5°C/min.

The cracking state analysis after different thermal cycles show that most of the cracks are indifferent from recycled aggregates but only by natural aggregates that compose them. The cracks are generated by the differential stresses between the cement paste and natural aggregates because of their incompatibility of deformation, while the new and old mortar behave as one unit because they are compatible.

We notice that the residual mechanical behavior of recycled concrete depends on the types of the aggregate. A significant loss of strength is observed between 300 and 450°C. The residual compressive strength of the recycled concrete BRM 0.3 at 20°C is 50 MPa, so a 40% less than ordinary concrete. The residual compressive strength increases up to 300°C. During heating, the migration of free water hydrates the CSH of cement paste and this increases the resistance. At 450°C the compressive strength is 25 MPa, while at 750°C is 7.2 MPa which is similar of the ordinary concrete BSC 0.3.

After heating to 750 °C, the calcite of the decarbonation of the lime (between 600°C and 650°C) reacts with ambient damp to form portlandite multiplying its volume of double. This increase in volume leads to the disintegration of concrete. No test of recycled concrete has erupted because of the high porosity which prevent the formation of high pressures inside the concrete. The tensile strengths and the residual elasticity modules are similar between recycled and ordinary concrete.

The analysis of the physical properties of recycled concrete show that for a W/C ratio of 0.3, the mass loss of concrete with recycled aggregates among the ordinary concrete is different up to 300°C. Beyond this temperature, the recycled aggregates and the ordinary ones have the same trend.

The maximum temperature difference between the surface and the center of the concrete specimen depends on how fast the water is to migrate outside. Two peaks are observed, they correspond to the evaporation of free water and bound water. The first peak corresponds to the end of the first phase of decomposition of CSH and the second peak
Thermical and mechanical behavior of recycled concrete aggregates and subjected to high temperatures

in the second phase of decomposition of CSH. The temperature difference corresponding to the first peak is higher for ordinary concrete, the recycled concrete contains higher porosity. Its higher porosity is even an advantage point because it allows a lower water pressure inside the concrete, so less/no spalling at high temperatures. The physical phenomena related to water appear to have a major influence on those specific thermal gradients. Accordingly, in terms of post-fire residual mechanical properties there are no limitations to the structural use of recycled concrete whether compared with conventional concrete.

In conclusion we can say that the recycled concrete due to its weaker mechanical properties (especially compression), it can not be used to build big buildings, or bridges, or works that require high mechanical strength. However, it can be used for houses or small buildings (like three floors), whereas the French norm (NF 206-1) gives the "allowed" mechanical properties for different kind of constructions.

The use of recycled aggregates in concrete provides a promising solution to the problem of the construction and demolition waste management, and the problem of scarcity of natural resources. Based on the properties of the recycled coarse aggregates and recycled concrete discussed in this paper, it is clear that the recycled concrete can be used in lower applications of concrete. The recycled coarse aggregates can be used to make normal structural concrete with the addition of flyash, condensed silica fume, etc.

Greater efforts are necessary in the direction of creating awareness, and relevant specifications to clearly demarcate areas where the recycled concrete can be safely used.
REFERENCE:


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[Xi L 2006] J. Xiao, P. Li, Qin W., Studio sui bond-slittamento tra calcestruzzo riciclato e armature, Giornale di Tongji University, 34 (2006), pp 13-16


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NORM:

EN 1097-6     Tests to determine the mechanical and physical properties of the aggregates, Part 6: Determination of volumic mass of the granules and the water absorption, CEN, 2008.


NF EN 13285     European standard that specifies requirements for mixtures with aggregates unprocessed natural, artificial and recycled, AFNOR, 2004.
