Universidade Federal do Rio de Janeiro
Escola Politécnica

OTIMIZAÇÃO DE MOLDES DE AREIA À BASE DE LIGANTES INORGÂNICOS PARA FUNDIÇÃO DE PEÇAS AUTOMOTIVAS EM ALUMÍNIO

Matheus Brozovic Gariglio

Projeto de Graduação apresentado ao Curso de Engenharia Metalúrgica da Escola Politécnica, Universidade Federal do Rio de Janeiro, como parte dos requisitos necessários à obtenção do título de Engenheiro.

Orientadora: Adriana da Cunha Rocha

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PROJETO DE GRADUAÇÃO SUBMETIDO AO CORPO DOCENTE DO CURSO DE ENGENHARIA METALÚRGICA DA ESCOLA POLITÉCNICA DA UNIVERSIDADE FEDERAL DO RIO DE JANEIRO COMO PARTE DOS REQUISITOS NECESSÁRIOS PARA A OBTENÇÃO DO GRAU DE ENGENHEIRO METALÚRGICO.

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Junho/2019

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Curso: Engenharia Metalúrgica

Algumas peças de fundição, como cabeçotes, são feitas com o uso de moldes de areia (misturas de areia e ligantes), que permitem a realização dos dutos internos dessas peças. Em um contexto ambiental cada vez mais restritivo, empresas de fundição estão desenvolvendo um novo processo de fabricação de moldes de areia à base de ligantes inorgânicos, visando reduzir suas emissões, em comparação à um processo com ligantes orgânicos. Por possuírem propriedades ainda pouco conhecidas pela indústria, existe a necessidade de adquirir meios de caracterização apropriados para esses novos ligantes. Neste trabalho, a motivação foi reformular os testes de caracterização (permeabilidade, flexão, resistência à umidade, demanda ácida da areia e deformação) utilizados pela indústria de fundição em areia na validação da qualidade de seus moldes. Onde foi possível destacar as características críticas do procedimento inorgânico, assim como as propriedades materiais dos moldes, reformulando os protocolos e especificações dos testes de laboratório da indústria de fundição em areia moldável, adaptando-a para um futuro onde serão apenas permitidos moldes de areia fabricados a partir de ligantes inorgânicos, visando diminuir drasticamente a poluição vinda dessa indústria, causando menos danos ao meio ambiente e melhorando a qualidade de vida de seus operários.

Palavras-chave: Ligante, Inorgânico, Fundição, Areia, Protocolo, Especificação, Propriedades, Umidade, Desmoldagem, Deformação, Permeabilidade, Ph

Abstract of Undergraduate Project presented to POLI/UFRJ as a partial fulfillment of the requirements for the degree of Metallurgical Engineer.

OPTIMIZATION OF FOUNDRY CORES BONDED WITH INORGANIC BINDERS IN THE AUTOMOTIVE INDUSTRY

Matheus Brozovic Gariglio

June/2019

Advisor: Adriana da Cunha Rocha.

Course: Metallurgical Engineering

Some castings parts, as cylinder heads, are made using cores (mixtures of sand and binders), which allow the production of the interior ducts of these parts. In an increasingly restrictive environmental context, foundry companies are developing a new coring process with inorganic binders, aimed at reducing their emissions, compared to a process with organic binders. As they have properties still little known by this industry, there is a need to acquire the appropriate characterization methods for these new binders. The purpose of this work was to redesign the characterization tests (permeability, bending, resistance to humidity, acid demand of sand and strain), usually used by the foundry industry to validate the quality of their cores. Thus, it was possible to highlight the critical characteristics of the inorganic procedure, as well as its material properties, reformulating the protocols and specifications of laboratory tests of the sand molded casting, adapting it to a future where only cores manufactured with inorganic binders will be allowed, aiming to drastically reduce pollution from this industry, causing less damage to the environment and improving the life quality of its workers.

Keywords: Binder, Inorganic, Foundry, Sand, Protocol, Specification Document, Properties, Humidity, Decoring, Deformation, Permeability, Ph

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LIST OF ABBREVIATIONS

Companies and Institutions

HA – Hüttenes-Albertus

AFS – American Foundry Society (also used as an index for sand fineness)

Characterization Tests

CaraMécas – Mechanical Characterization

ADV – Acid Demand Value

Materials and processes

Core – Sand mold

Coring – Core manufacturing

Inorganic cores – Foundry sand cores bonded with inorganic binders

VOC – Volatile Organic Compound

Parameters

T - temperature

s – seconds

min - minutes

h – hour

t – time

daN – decanewton

RH – relative humidity

Equipment used

DMEA – Dimethylethanolamine

LVDT - Linear Variable Differential Transformer

1. INTRODUCTION

Some foundry casting parts, as cylinder heads, are made using cores (mixtures of sand and binders), which allow the production of the interior ducts of these parts. In an increasingly restrictive environmental context, many foundry companies are developing a new coring process with inorganic binders, aiming to reduce its pollution effects when compared to a process with organic ones. These new silicate binders consist of sodium silicate and a mineral powder, allowing a substantial reduction of VOC emissions throughout the foundry process. However, their material properties are not well known in the industrial sector. Hence, in order to acquire the appropriate characterization means for these new materials, one of the main foundry companies in France, Montupet, desired to review the laboratory test protocols and specifications used in the validation of the binder’s quality, as the characteristics of a good organic core are not necessarily valid in the case of the inorganic ones.

Foundry cores must meet several requirements, particularly in terms of mechanical properties, humidity resistance, acid demand of sand, as well as being sufficiently resistant to allow casting and, thereafter, to degrade easily, making possible their decoring. The purpose of this work was to highlight the critical characteristics of the inorganic cores, as well as to write the laboratory test protocols and specifications to adapt them to this new process, allowing foundry companies to better integrate into a world that increasingly considers the environment’s quality and the employees’ health.

Characterization tests such as, permeability, bending, humidity resistance, acid demand of sand and strain, usually used by the foundry industry to validate the cores’ quality, were redesigned in this study. The critical characteristics of the inorganic procedure, as well as its material properties, were then determined, leading to the reformulation of protocols and specifications of the sand molded casting.

2. LITERATURE REVIEW

2.1 FOUNDRY AND DIFFERENT CASTING PROCESSES

Foundry is a metal manufacturing process that involves casting a metal or a liquid alloy in a mold to reproduce, after cooling, a given piece (inner and outer shape) by limiting as much as possible the subsequent finishing work. The advantages of casting in comparison to other processes, as machining, joining, additive manufacturing, forming and molding, are:

- Possibility of producing metal parts with very high volumes;
- High complexity, impossible to be obtained by machining, for example;
- High productivity;
- Difficulty in welding some alloys.

The techniques employed in foundry depend on the molten alloy, dimensions, characteristics and quantities of parts to be produced. It is an industry highly dependent on the acquiring sectors: automobile, iron and steel, material handling equipment, industrial equipment, electrical equipment, aeronautics, weapons, etc.

There are several kinds of foundry processes, such as Investment Casting, Die Casting, Centrifugal Casting, Shell Molding, Squeeze Casting, Green Sand Casting and Sand Molded Casting, but this study is focused on the last one (CUENIN, 1999).

2.2 PROCESSES FOR MANUFACTURING A CYLINDER HEAD BY SAND MOLDED CASTING

The cavities of a cylinder head ensure the mixing of gasoline and air, which allows the combustion and circulation of other fluids providing lubrication of the mechanical elements and cooling of the engine. According to the gasoline or diesel model, the cylinder head has different manufacturing processes.

The dimension of a cylinder head has a direct influence on the engine performance, which is the reason why development lies at the heart of Montupet's know-how. In order to better understand the manufacture of a pre-machined cylinder head, here is a description of the manufacturing process, as shown in Figure 1 and thoroughly explained in the following sections (CUENIN, 1999).

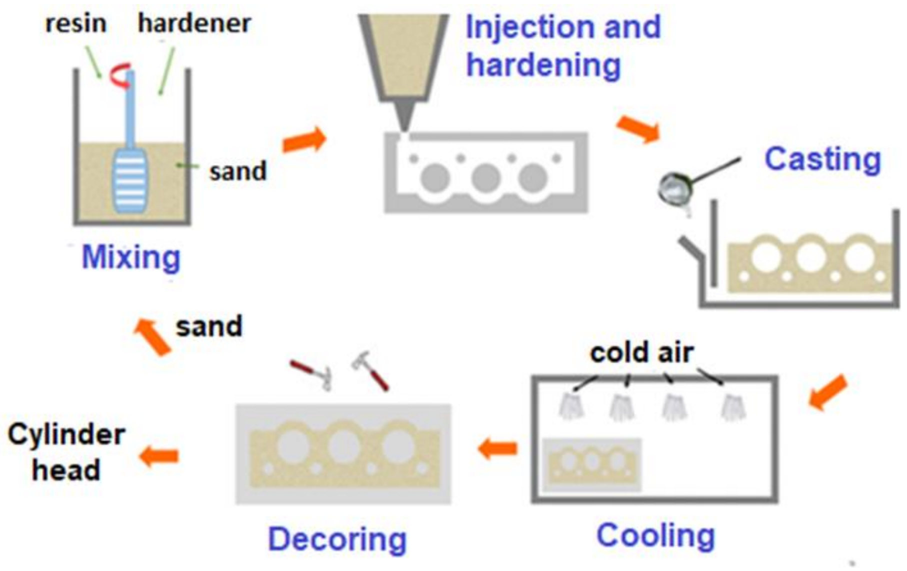


Figure 1 - Simplified diagram of the processes for manufacturing a cylinder head [1]

2.2.1 THE CORING PROCESS

Coring is a technical term for making sand cores. They allow the manufacture of the internal hollow parts of a cylinder head, thus achieving complex inner cavities. To give an idea, the volume of sand used is almost twice as high as the aluminum volume of the raw cylinder head. The process used in the foundry industry for the moment is the organic "cold box" (Figure 2). In contrast, in an increasingly restrictive environmental context, foundry companies are developing a new process for "hot box" coring with inorganic binders, aimed at reducing their emissions (GARAT, 2013).

[1] Source : Company's confidential report

The desired core properties are:

• Enough mechanical strength before casting and weakness after, to allow breakage;

• Refractoriness;

• Permeability;

• Dimensional accuracy and clean surface finish;

• Low cost and possibility of sand reclamation.

Figure 2 – Complete set of cores (organic binder)[2]

2.2.1.1 MIXING

The cores are composed of sand calibrated AFS 45 (fineness index of sand grains) by adding organic binders (before this work) or inorganic (after this work). Raw materials are constantly monitored in the sand laboratory to ensure the quality of incoming products in foundry companies. The quality of the core is an important factor to meet a precise specification (FEDORYSZYN, 2013).

2.2.1.2 INJECTION AND HARDENING

The reaction of the binder on the sand is a polymerization, enabling the creation of hardened binder bridges between the grains of sand, which makes it possible to obtain a solid assembly. In the case of:

[2] Source : Company's confidential report

- **Organic binders**: the cores are obtained by putting the mix in a "box of cores" inside a coring machine that injects it into a metallic mold and gases the mix with amine (DMEA) → “cold box” process;
- **Inorganic binders**: the polymerization reaction is very long at ambient temperature; therefore, cores are produced on a coring machine that injects the mix into a metallic mold and gases the mixture with hot air, to catalyze the polymerization → "hot box" process.

The different cores are assembled and sometimes glued to form, what is called, a core cluster (JASSON, 1999).

2.2.2 CASTING AND COOLING

Foundry companies usually do not elaborate their own alloys, instead, they buy metallic ingots containing the desired compositions. However, during melting, it is necessary to control the metal’s composition, making some adjustments to achieve the desired values. Then, impurities are removed and the liquid metal is stored, at about 730°C, in holding furnaces for a specific time until the casting step.

When the metal is ready, the liquid aluminum can be casted by (Figure 3):

- **Gravity:** the ladle pours the liquid metal into a duct that feeds the mold;
- **Low pressure:** the holding oven is below the mold, and the aluminum is pushed by pressure from the oven to the mold;
- **Tilted:** process with tilting of the mold-ladle assembly during casting.

The cylinder heads are then cooled in ambient air and using wind tunnels.

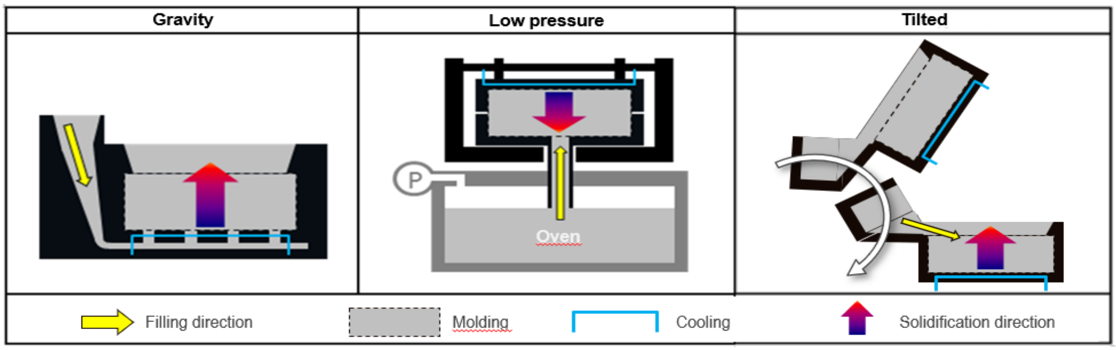


Figure 3 – Molding processes[3]

2.2.3 COMPLETION

The cylinder heads continue to cool down before reaching the completion zone. This sector will eliminate parts that do not fit into the final design. Cutting machines remove the feed descents from the workpiece as well as the centrifugal weights (Figure 4).

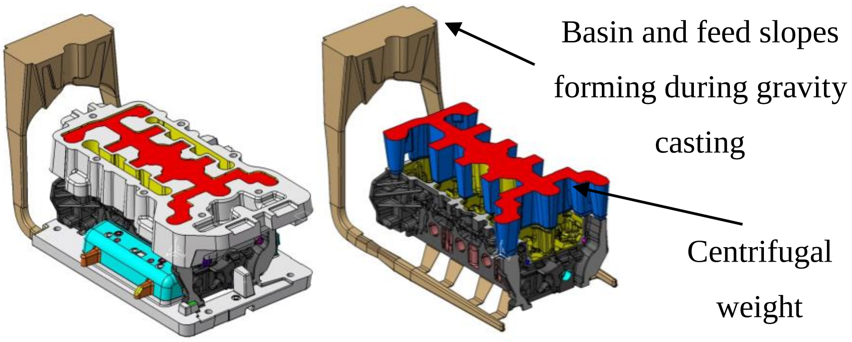


Figure 4 - Molding with the whole of the cluster and after the decoring is done[4]

2.2.4 DECORING AND PRE-MACHING

The last step is the decoring, where machines will hammer the cylinder heads and vibrate them to break and extract all the cores contained inside.

[3] Source : Company's confidential report
[4] Source : Company's confidential report

In order to finalize the manufacturing process, the parts will undergo a battery of operations before shipping. Parts with higher added value are heat-treated to increase mechanical performance. The final phase is the pre-machining, a series of surfacing to meet the customer specifications. Washing and marking steps ensure cleanliness and traceability, respectively. A final visual check identifies the presence of all holes and dimensional compliance and, finally, the cylinder heads are palletized before shipping.

2.2.5 RECLAMATION

On account of environmental problems and economic constraints, cores already used in production pass through a last step, the sand reclamation. It consists of a crushing of the cores, then a heat treatment at 520°C (in the case of organic binders) to remove the remaining binders, and finally a sieving step, where the remaining aluminum is separated, and the particle size is controlled. The reclamation yield is about 95%.

2.3 THE INORGANIC PROCESS

The main steps of the inorganic process remain the same in comparison with the organic one (section 2.1). However, there is a great difference in the coring step, due to the completely different chemistry related to the polymerization of the inorganic cores, therefore the process is strongly impacted. A hot air gassing process is required instead of the DMEA (see section 2.2.1.2), and a very important precaution concerning the humidity uptake of the cores during their storage.

Inorganic binders consist of two parts (SORO, 2015):

- **Liquid:** Sodium silicates in modified solution with adjuvants of different natures (Figure 5);

Figure 5 – Modified solution of sodium silicate[5]

- **Powder:** additives, composed of different oxides (mainly, and in order: Si, Al, Zr and Fe), as shown in Figure 6.

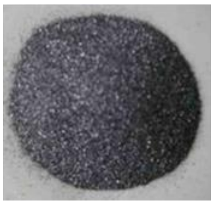


Figure 6 **-** *Mineral powder (natural + synthetic)*[6]

2.3.1 SODIUM SILICATES (LIQUID)

The general formula is: x Na_2O, y SiO_2, z H_2O. In the foundry industry, they are present in the form of solutions, obtained by dissolving in water in basic environment of sand or glass, hence the reason of their basic pH (LUCAS et al, 2011).

The polymerization of the binder is obtained by polycondensation of the silicates. This reaction is catalyzed by an increase in temperature, due to the removal of water from the solution, then the charged colloids gradually get closer and combine to form a silicate glass (inorganic bridges), as shown in Figure 7 (TOHOUE et al, 2012a).

[5] Source : Company's confidential report
[6] Source : Company's confidential report

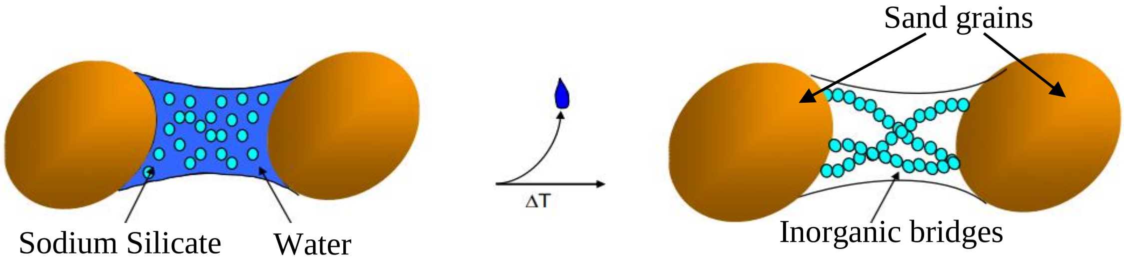


Figure 7 - Diagram of drying and formation of inorganic bridges[7]

2.3.2 ADDITIVES (POWDER)

The additives can be added directly to the sand or premixed with the inorganic binder, which are then added to the sand by a conventional dosing system. They are composed largely by mineral elements, consisting of silica or silico-aluminous submicron (smoke). Their roles are to improve (FEDORYSZYN et al, 2013):

- The flowability of mixed sands to facilitate shots;
- The increase of core resistance during gassing with hot air;
- Stability to the humidity uptake;
- The breakage and decoring by their intervention in the chemical process of consolidation.

3. MATERIALS AND METHODS

3.1 RESEARCH METHODOLOGY

The aim is to approach the problem by analyzing the current characterization tests implemented in the foundry industry. Hence, to create a project plan, all the factors that may influence those tests must be varied, in order to find out the critical characteristics of inorganic cores, thus optimizing the existing tests and proposing new ones.

[7] Source : Company's confidential report

3.1.1 ISHIKAWA DIAGRAM – ADDRESSING THE PROBLEM

During the study of inorganic cores, it was necessary to identify all the factors that could cause problems during the manufacture the cylinder heads. The Ishikawa Diagram (5M method) has been implemented because it allows the identification and classification of the causes resulting in an effect (Figure 8):

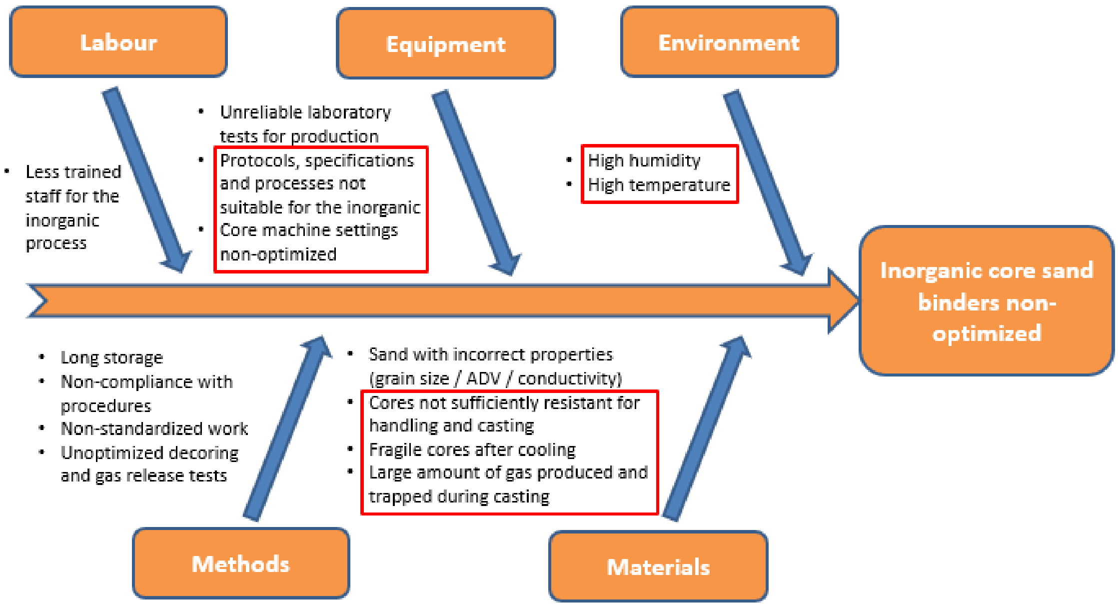


Figure 8 – Ishikawa Diagram for the problem of cores bonded with inorganic binders

A major problem of the inorganic process concerns the main difference that exists between the organic and inorganic cores: the loss of mechanical properties due to humidity uptake. Since in many countries, as China, the humidity is much higher than in France, about 80% RH during summer, a thorough study of the mechanical behavior of cores as a function of their time of exposure to humidity is necessary to optimize their resistance, fragility and thermal degradation.

Another factor that could lead to coring problems is the adjustment of the parameters of the coring machine, as there are several different parameters that can affect the core properties, such as cooking and gassing times, temperature of the box, shot pressure, among others.

The specifications for sand and cores had not been yet updated for the new inorganic process. They give the expected specifications for the raw materials, which means the validation tests to be performed and the description of the range of acceptable results for Montupet. The aim of this study is to list the tests to be performed, to adapt or create experimental protocols, and to define requirements for each test, considering the specificities of cores bonded with inorganic binders.

3.1.2 ANALYSIS OF CHARACTERIZATION TESTS IMPLEMENTED IN FOUNDRY COMPANIES

First, an analysis to find out what were the usual tests for characterization of sands and cores in foundry companies has been performed, as well as the new tests that could be implemented, along with their reasons for being so. The result of this analysis is shown below (MARTINEZ, 2015):

- **Classic sand characterization tests:**
    - **Granulometry:** a high content of fines decreases the storage time and the mechanical strength of the cores;
    - **ADV (Acid Demand Value):** A high ADV can trigger the polymerization reaction of the organic resin during mixing. In the case of inorganic binders, measures the degree of pollution of the reclaimed sand;
    - **Conductivity:** analyzes a possible sand pollution.

- **Classic core characterization tests:**
    - **CaraMécas (Mechanical Characteristics):** the cores must be sufficiently resistant after ejection, during handling and casting;

- **Humidity resistance:** high humidity drastically reduces the storage time and the resistance of inorganic cores;
- **Decoring:** checks for easy removal of cores from the metallic piece after casting.

- **New tests to be implemented in this work:**
  - **Permeability:** verifies the ease with which gases, generated during casting, pass through cores and not be trapped, and if the gassing of cores during coring is done completely;
  - **Strain:** measures the strain at maximum stress because highly deformable cores can cause dimensional problems on the cylinder heads, and very fragile cores easily break with handling.
  - **pH:** as the ADV, also measures the degree of pollution of the reclaimed sand. Nevertheless, its seems to be a cheaper and faster alternative.

As a consequence of this analysis, along with the 5M method, the order of priority of the tests to be optimized has been established in 5 different phases:

1. **CaraMécas + Humidity resistance:** direct impact of humidity and storage time on the core properties, one of the main problems of the inorganic process;
2. **Strain:** possibility to give several new information on the mechanical properties of the cores (strain at maximum stress, yield strength, Young's modulus...);
3. **ADV + pH:** the requirement of the specifications, for the previous organic binders, was very severe for inorganics without necessity, the influence of a pH test has been required, as well as the need for a quality analysis of the sand reclamation;

4. **Permeability:** a test that did not have a protocol for characterization and was important to be studied, on account of its influence during casting on the quality of cylinder heads;

A summary of all the tests that have been optimized or implemented during this work it is shown in Table 1.

Table 1 – Summary of all the tests that have been optimized or implemented

	Classic characterization tests	Tests to be optimized	New tests to be implemented
Sand	Granulometry		pH
	ADV	✓	
	Conductivity		
Core	CaraMécas	✓	Permeability
	Humidity Resistance	✓	Strain
	Decoring		

The granulometry test has not been revised because the AFS 55 reference sand will always be used to carry out the tests in Montupet. The conductivity test validates the non-pollution of the sand, and the change of process does not call into question this test. And finally, decoring is a very complex test to be simulated in a lab, so it has been concluded that a "mini-decoring machine" should be developed to optimize this test completely, so it has not been fitted into the planning of this work.

3.1.3 MATERIALS AND PARAMETERS OF CORE MANUFACTURING

The inorganic process used at Montupet is the Cordis one, developed by Hüttenes-Albertus, which consists of a completely two-component inorganic system, composed by a modified silicate solution (Cordis), and the additive (Anorgit), as described in section 2.3.

The raw materials and their quantities recommended by Hüttenes-Albertus for conditions of high humidity, therefore applied at Montupet, are as following ones:

- **Silicate solution (liquid):** Cordis A or Cordis B (the use of one or the other reference will depend on the geometry of the cores. In the laboratory, the former was used), with a rate of addition of less than 3% by weight of sand;
- **Additive (powder):** Anorgit A, with a rate of addition of 1% by weight of sand;
- **Sand:** siliceous sand with AFS 55 (fineness index).

Figure 9 is a summary of the manufacturing parameters, of the inorganic cores, used before the start of the study. All the parameters are adjustable, however it was decided to focus on the cooking and gassing times, because a simulation done previously in the company has shown that the gassing is more important than the temperature of the box in the polymerization. Also, it has been learnt that Montupet's competitor and supplier were performing their tests with cooking and gassing times of 30 seconds.

In addition, it was decided to maintain the temperature of the coring box, because according to the literature, a temperature regulation between 140-200°C is necessary (LUCAS et al, 2011):

- **Temperatures < 140°C:** increase the core's setting time (polymerization) and decrease its storage stability;
- **Temperatures > 200°C:** lead to a degraded surface condition of the core, due to reactions of binder additives and dehydration.

The shooting pressure, as well as the shooting time, were maintained as they are recommended by the supplier and they have been considered good by the coring team at

Montupet. Finally, the temperature of the hot air could not be increased because of technical constraints.

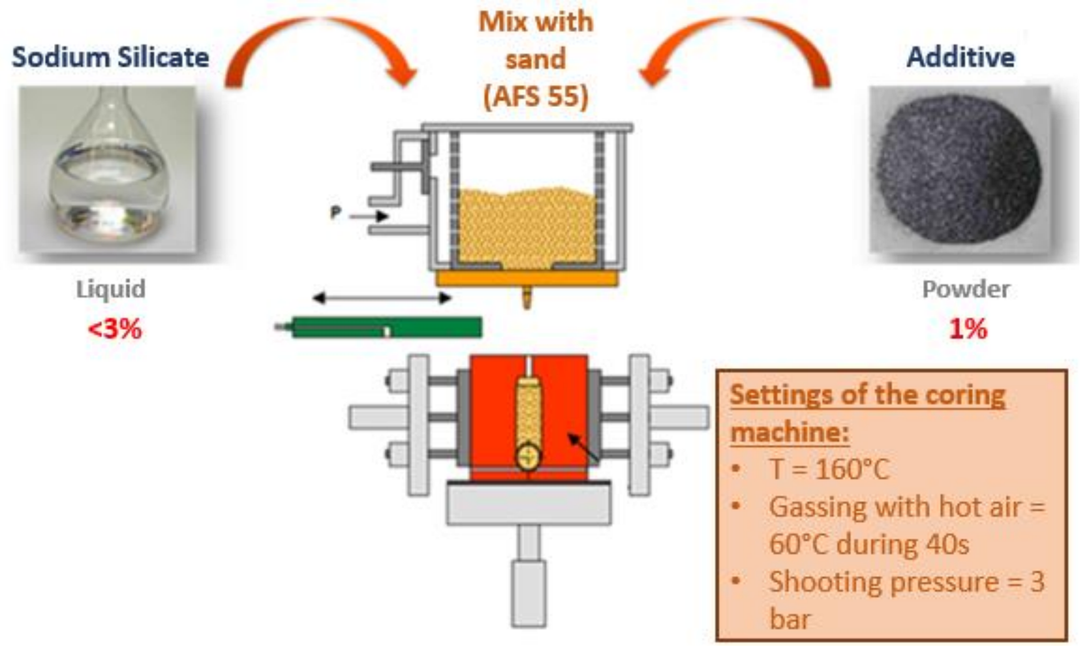


Figure 9 - Diagram of the core manufacturing, at the beginning of the study, in the Montupet's laboratory[8]

3.1.4 INITIAL TEST PLAN

After analyzing the Ishikawa Diagram of the characterization tests and the core fabrication parameters at Montupet, it was decided to start the work by modifying the storage conditions of the cores, analyzing their mechanical behavior as a function of time. Then, the influence of the manufacture of core specimens on their mechanical properties has been analyzed, more particularly the cooking and gassing times. In this way, an initial test plan for CaraMécas and Humidity Resistance has been chosen, where a different parameter has been changed by each test, in order to notice its single influence on the core's mechanical properties (Table 2).

The temperatures and humidity of the tests are the following ones, and they were chosen because:

[8] Source : Company's confidential report

- **25°C and 40% RH -** Sand laboratory conditions in France (Laigneville)
- **25°C and 80% RH -** Average conditions of the city in China (Wuxi) where there will be implanted a new Montupet's foundry industry
- **45°C and 80% RH -** Extreme conditions of Wuxi
- **45°C and 40% RH -** Check the influence of the temperature on the cores

Table 2 – Initial tests plan (changed parameters are shown in red)

Test Number	Storage T (°C)	Storage RH (%)	AFS	Cooking and gassing times (s)	Changed parameters
1	25	40	45	40	Standard
2	25	80	55	40	RH
3	45	80	55	40	RH and Temperature
4	45	40	55	40	Temperature
5	25	40	55	40	AFS
6	25	40	45	10	Cooking and Gassing
7	25	40	55	10, 15, 20, 30	Cooking and Gassing

The AFS of the first test is 45 because, at the beginning of the experimental work, Montupet still had not received the AFS 55 from the supplier. However, it has been shown with the test n°5 (section 4.1.5), that there is no great influence of this change on the results obtained.

As the tests were being done, other parameters have been changed and new tests have been implemented. However, these will be explained during this study, for a clearer explanation of the reasons for each one.

3.2 EXPERIMENTAL PART

3.2.1 MECHANICAL CHARACTERIZATION AND HUMIDITY RESISTANCE

The mechanical characterization (CaraMécas, as named by the company), together with the humidity resistance, are two of the main tests carried out to characterize a core bonded with inorganic binders. They highlight whether the core will be sufficiently resistant (after the ejection, during handling and casting), as whether it can withstand high humidity for hours.

These tests consist of producing bar-type specimens and performing 3-point bending tests, in different storage times and conditions. The steps of this process are better explained in the following sections (AMERICAN FOUNDRY SOCIETY, 2015).

3.2.1.1 MANUFACTURE OF 3-POINT BENDING TEST SPECIMENS

First, to carry out the various characterization tests, a "Laempe" coring machine (Figure 10) has been used, where bar-type specimens have been manufactured.

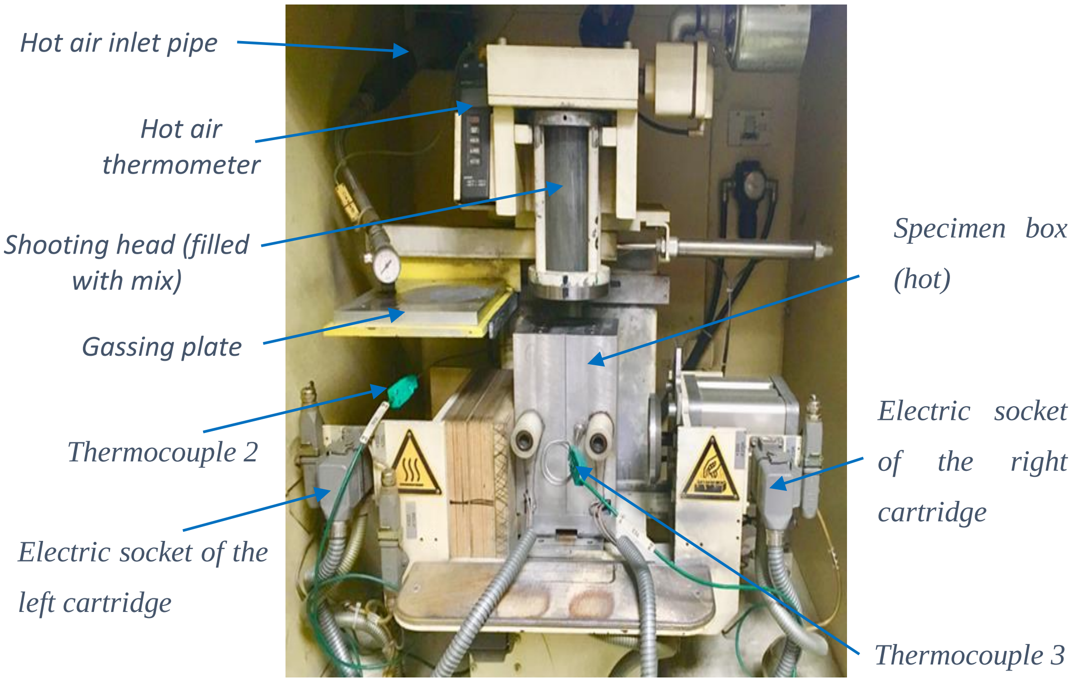


Figure 10 – "Laempe" Coring Machine with its accessories

The mix, placed in the shooting head, has been always prepared before the tests, prepared with a mixer, as show in Figure 11:

- **Liquid:** Cordis A (< 3% by weight of sand);
- **Powder:** Anorgit A (1% by weight of sand);
- **Sand:** siliceous sand with AFS 55 (usually 4 to 5 kg).

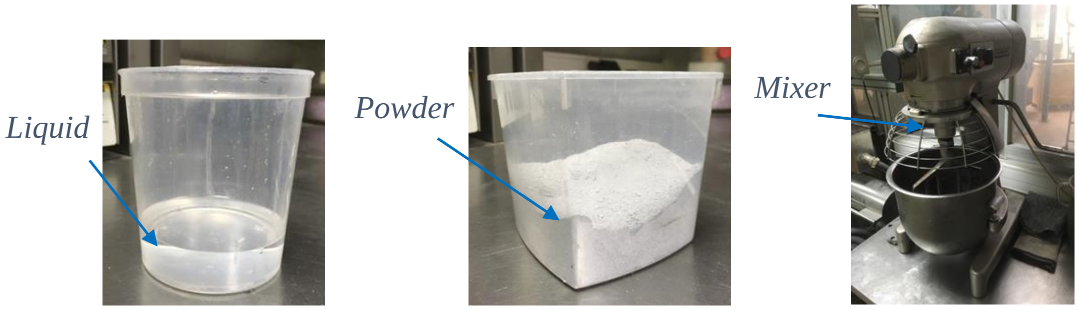
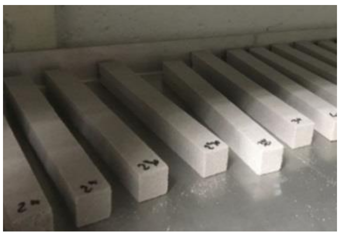
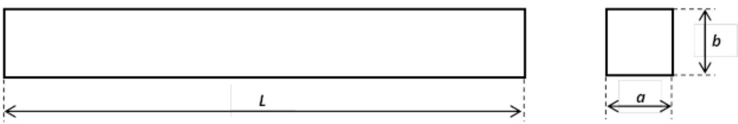


Figure 11 - Raw materials for the core manufacturing

The bar-type specimens are parallelepipedal in shape, with a square section. Dimensions are depicted in Figure 12:





L	*a*	*b*
225 mm	22,4 mm	22,4 mm

Figure 12 - 3-point bending test specimens

3.2.1.2 COOLING INSTRUMENTATION

As the specimens come out of the hot box at temperatures above 100°C, it has been performed a core cooling instrumentation, immediately after its manufacture, by putting a thermocouple inside the specimen and connecting it to an acquisition center, as depicted in Figure 13. Figure 14 presents the result, cooling curve, for this process.

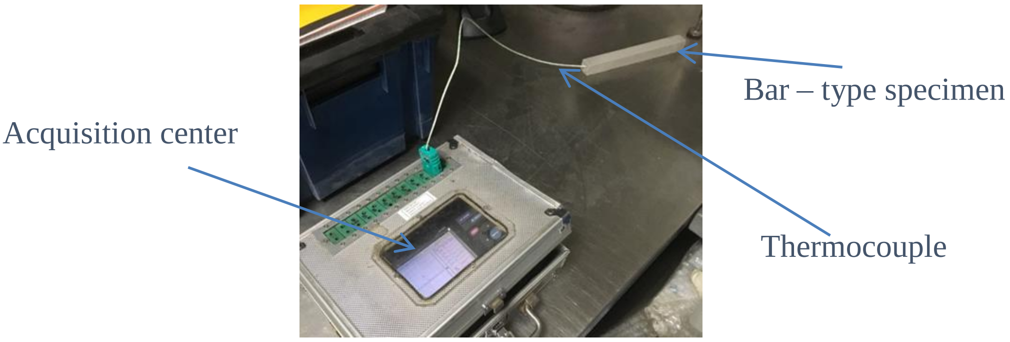


Figure 13 - Instrumentation of a bar-type specimen used in the bending tests

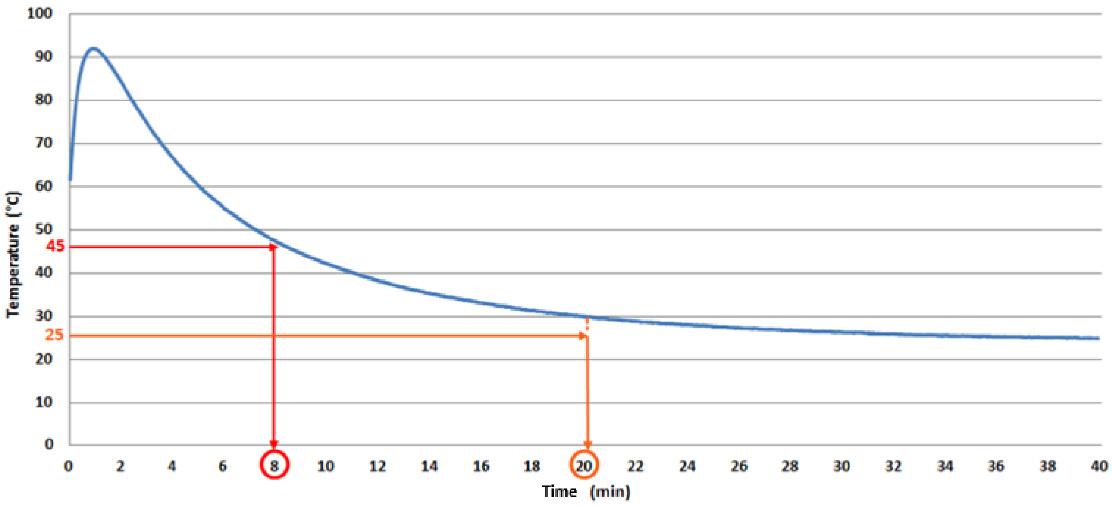


Figure 14 - Cooling of specimen (cooking and gassing times of 40s and temperature of the specimen box at 160 ° C)

After the analysis of this curve, it was noticed that it takes 8 min and 20 min to put the cores in the climatic chamber set at 45°C and 25°C, respectively.

3.2.1.3 BENDING TEST UNDER DIFFERENT TEMPERATURE AND HUMIDITY CONDITIONS

After the manufacture of the cores, a SIMPSON 3-point bending machine (Figure 15) has been used to carry out the CaraMécas and humidity resistance tests.

Figure 15 – SIMPSON 3-point bending machine

For tests 1 to 4 of the initial plan (Table 2), 14 storage times (Figure 16) have been chosen in order to analyze the behavior of the specimens according to the different temperature and humidity conditions, as well as to choose the optimal times to break (through the bending test) the specimens during the characterization of a new binder. Two specimens per time have been tested in this initial test.

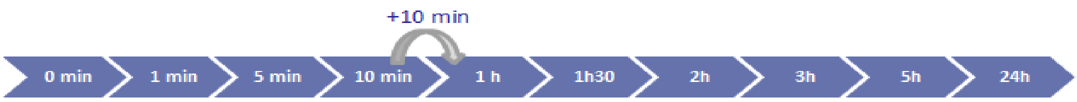


Figure 16 – Different storage times of specimens

3.2.1.4 MASS VARIATION (HUMIDITY UPTAKE)

Concurrently with the bending tests, mass variation tests, using a laboratory scale (with a precision of 0.001g), have been performed between the time that the specimen is removed from the box and the 3-points bending test.

3.2.1.5 VARIATION OF GASSING AND COOKING TIMES

Given that the supplier (HA) and the competitor (Nemak) used a cooking and gassing times of 30 seconds to carry out their tests, various tests have been carried out in order to qualitatively analyze the effect of the cooking and gassing on the cores

(DOBOSZ et al, 2011). First, 5 cores (10, 15, 20, 25, 30 seconds of gassing) have been manufactured and cut in half, to analyze the cooking conditions in the inner part.

3.2.1.6 NUMBER OF SPECIMENS (REPEATABILITY)

The number of specimens has been sought to be optimized, in order to find a good compromise between the time required to perform the tests and the representativeness of the results. For this, mechanical tests have been carried out in different conditions (gassing and storage) on 2, 4 and 6 specimens, each time, in order to compare their results in precision and accuracy.

3.2.1.7 VARIATION OF THE GRANULOMETRY OF THE SAND

As explained in section 3.1.4, the AFS of the test n°1 was 45, because, at the beginning of the work, the AFS 55 was not available. Tests to compare the two AFS and analyze the influence of granulometry, on CaraMécas test, have taken place.

3.2.2 IMPLEMENTATION OF A NEW STRAIN TEST

In order to better understand the mechanical properties of the cores, a force and a displacement sensor have been implemented on the bending machine. The main information that has been desired to be extracted from this test was the strain at maximum stress and the stiffness of the cores (elastic modulus). Indeed, cores bonded with inorganic binders have the particularity of being very fragile, which leads some steps to be delicate (manipulation and assembly, for example). On the other hand, it was noticed that these cores could be deformed within ten minutes after ejection of the box, which could generate non-dimensional metallic parts. These two characteristics motivated us to set up this new characterization test.

After obtaining the forces and displacements values, directly by the implementation of the respective captors, the stress and strain, in 3-point bending tests, are given by the following formulas (VARGAS, 2018):

$$\sigma\left(\frac{daN}{cm^2}\right) = \frac{3FL}{2bh}$$

$$\varepsilon\left(\frac{mm}{mm}\right) = \frac{6fh}{L}$$

Where,

- **F** = Force (N)
- **L** = length between the 2 supports (mm)
- **b** = specimen length (mm)
- **h** = specimen height (mm)
- **f** = deflection (mm)

3.2.2.1 MATERIALS AND ASSEMBLY

After a technical drawing and the desired modifications on the available parts have been carried out, the assembly of the test was finalized: the force sensor is on the right part of the support (inside the hull-knife device), while the displacement sensor (LVDT) is on the left (see Figure 17).

Figure 17 – Assembly of force and displacement sensors on the bending machine

Electronical and mechanical adaptations had to be made to assemble properly and to obtain a rigid system, which does not disturb the measurement of the sensors.

3.2.2.2 CALIBRATION AND VALIDATION TESTS

A calibration of the sensors has been performed in comparison with the Simpson measurements, using a video camera, as the SIMPSON machine does not have a data recording system. Some results of the validation tests are shown in Figure 18:

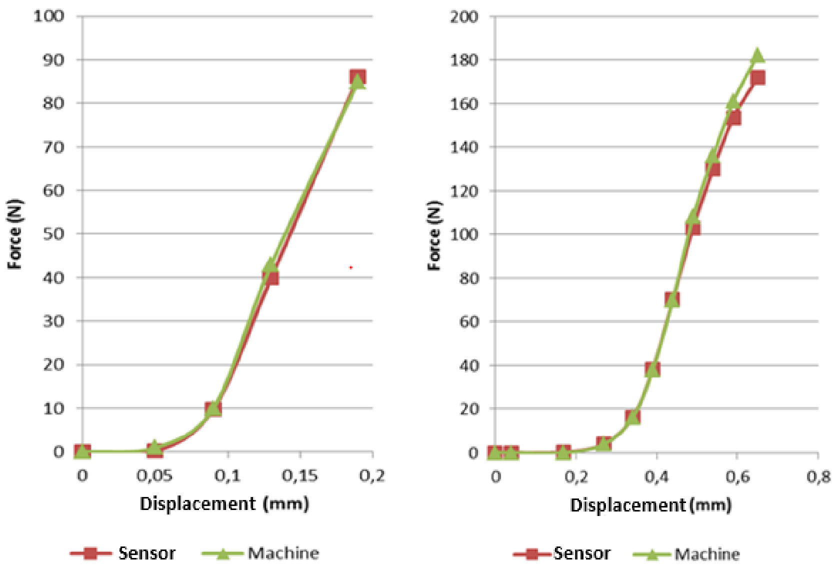


Figure 18 - Results of the comparison between the values of the machine and the sensor

The results were very close, so it can be stated that the sensor measurements have been validated. In total, 60 stress-strain curves have been recorded, where the relative errors between them and the Simpson machine were, on average, less than 5%.

Then, tests have been carried out with 16 specimens on 8 different conditions of cooking and gassing times (15s and 30s), storage (0h, 2h, 5h) and humidity (40% and 80% RH), as determined in section 3.2.1.3.

3.2.3 ADV (ACID DEMAND VALUE) AND PH OF THE SAND

In order to measure the quantity of alkali metals and various impurities contained in foundry sands, as well as to control the quality of the reclamation, ADV tests are systematically carried out at many industries around the world (AMERICAN FOUNDRY SOCIETY, 2015).

Using an automatic titrator, 30 g of sand are first mixed with 30 ml of water and 30 ml of hydrochloric acid (HCl) in a beaker, then the titrator adds sodium hydroxide (NaOH), dropwise, until the pH of the sample reaches 7. The same procedure is carried out for a blank (without the sand) and the ADV of the sample is the difference, in ml, between the acid initially poured into the sample and the remaining acid (not consumed by the sand) after 5 minutes of stirring (Figure 19).

$$ADV_{(30)} = V_i(1 - \frac{V_E}{V_b})\text{, where}$$

- **V_i** (ml) = Volume of HCI 0.1 poured into the sample (30 ml)
- **V_E** (ml) = Volume of NaOH 0.1 N poured into the sand sample to reach equivalence
- **V_b** (ml) = Volume of NaOH 0.1 poured into the white to reach pH 7

Figure 19 – ADV test material

The ADV test has been initially planned to detect external pollution of the sand, like limestone residues from the soil, for example. However, inorganic reclamation has a very strong impact on the ADV, due to the nature of the inorganic binder, which is not completely eliminated during this process. In this way, the sand measurement of inorganic reclamation is no longer representative of impurities, but of binder residues. So, the usual specifications are no longer valid, and the protocol had to be updated (ASK, 2017).

Some pH tests have also been carried out, at the same time, because, according to ASK (one of the binder suppliers), it is necessary to carry out 2 different tests on foundry sands, ADV and pH, in order to check their quality. If the impurities present in the sand are soluble in (Figure 20):

- water, they will change the pH;
- diluted acid, they will modify the ADV.

Sand sample	pH	ADV
High-purity silica sand	7.16	0.1
0.02% added sodium hydroxide	11.1	2.5
0.2% added calcium carbonate	9.5	27.5

Figure 20 - Influence of different types of impurities on ADV and pH tests (Source: ASK)

In some tests carried out at Montupet previously, it has been shown that the ADV of a sand reclaimed from inorganic cores decreases with the increase in the heat treatment's temperature, because of the vitrification of the binder. So, 3 heat treatments have been carried out in order to obtain reclaimed sands with different ADV values.

For the inorganic process, ADV becomes a test of "dosage" of the residual amount of active binder, being necessary to know the threshold impact on CaraMécas. In order to verify this influence and the relevance of the implementation of a pH analysis, the test plan below has been carried out (Table 3):

Table 3 – Test plan for sand testing

Cooking temperature (°C)		0		200		400		600	
Test		1	2	1	2	1	2	1	2
ADV	White								
	Sand								
pH	White								
	Sand								
Bending test at ambient conditions (25°C et 40% HR)	0h								
	2h								
Bending test at humid conditions (25°C et 80% HR)	2h								
	5h								

2 specimens per pH and ADV test

CaraMécas

4 specimens per condition

Humidity resistance

A sand reclamation was simulated by completely grinding 10.5 kg of specimens, into powder, and putting 3.5 kg in the oven at 3 different temperatures, for 2 hours, in order to achieve the desired heat treatments in the plan above. Then, the reclaimed sand was mixed with 15% of new sand and the tests of ADV/pH have been carried out on these mixes, while CaraMécas and Humidity Resistance have been performed on cores manufactured from the mixes mentioned. The pH has been determined directly by adding 90 ml of the demineralized water with 30 g of sand, according to the American Foundry Society tests (STAUDER et al, 2018).

3.2.4 PERMEABILITY

Permeability is a physical feature that represents the ease with which a material allows fluid transfer through a connected network, playing an important role in the cores used for cylinder heads production. Indeed, the permeability of the cores must be enough to facilitate cooking (air gassing), and degassing during casting. On the other hand, it cannot be too high to limit the phenomena of watering: penetration of the liquid metal between the grains of sand, which gives a rough metallic surface and with inclusions of sand (TOHOUE et al, 2012b).

At Montupet, permeability is measured using a permeameter (Figure 21):

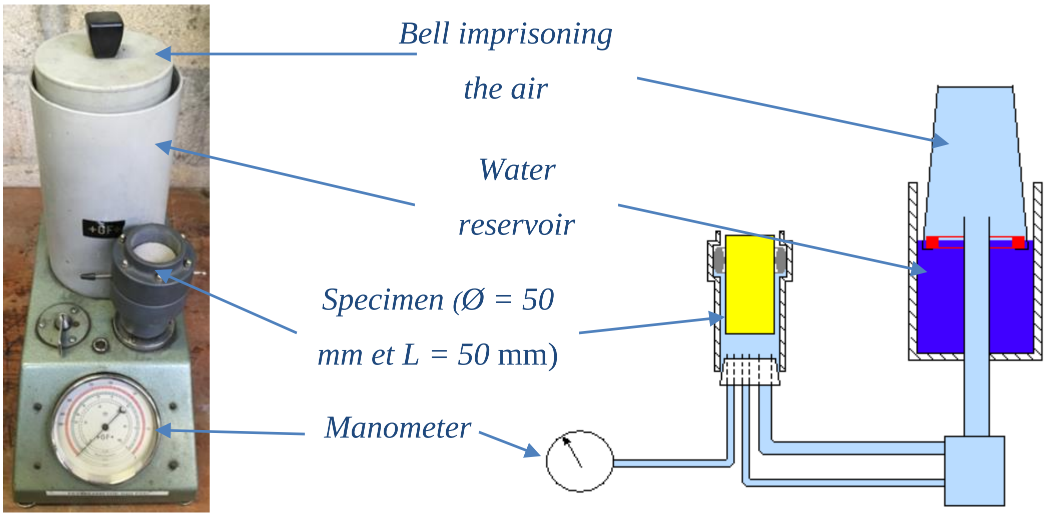


Figure 21 - Permeability test (with its diagram on the right)

Once the specimen is positioned, the bell is raised to fill it with air. It descends under its own weight and puts pressure on the air contained in the circuit. The relative pressure of the air under the specimen is measured, which depends directly on its permeability. The manometer present on the permeameter gives the permeability index in Darcy (1 Darcy = 0.97^{-12} m²).

Three specimens have been produced in the same way explained in section 3.2.1.1 (except the specimen hot box mold, which has been changed), for each of the 6 storage conditions (2h, 5h), cooking and gassing times (15s, 30s) , and humidity (40% RH and 80% RH).

4. PRESENTATION AND ANALYSIS OF RESULTS

4.1 MECHANICAL CHARACTERIZATION AND HUMIDITY RESISTANCE

4.1.1 BENDING TEST UNDER DIFFERENT TEMPERATURE AND HUMIDITY CONDITIONS

The results of the evolution of the mechanical resistance as a function of time and storage conditions, as well as the break times used in the characterization protocols, before and after this study, are shown in Figure 22:

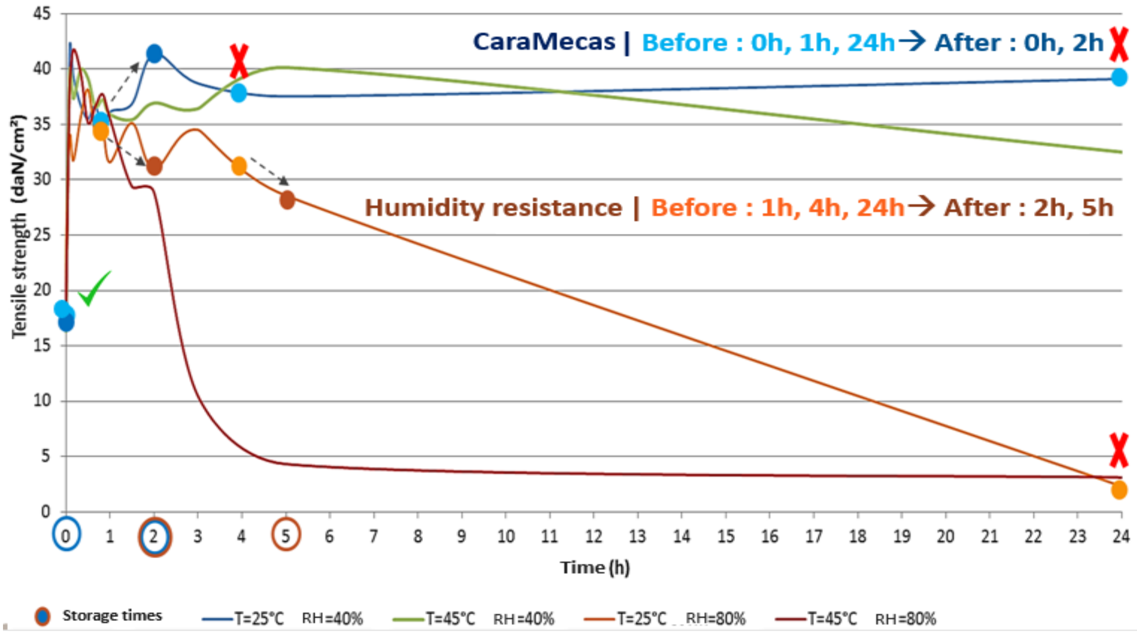


Figure 22 - Mechanical behavior as a function of time and storage conditions (with break times)

When analyzing the curves, it is clear that the break times, chosen before, were not optimal to carry out the characterization tests, except at 0h (after ejection). The changes that have been made, as the reasons of being so, are shown in Table 4:

Table 4 - Break Time Changes for CaraMécas and Humidity Protocols

Storage times and conditions		Why?
Before	**Now**	
Ambient: **0h - 1h - 24h**	**Ambient:** **0h - 2h**	• **Keep 0h :** simulates the resistance after ejection; • **Change 1h → 2h :** the difference between ambient and wet storage is clearer at 2h, and curves are more stable at that storage time; • **Change 4h → 5h :** The effect of humidity is more visible after 5h (at 4h it is comparable with 2h); • **Delete 24h :** not characterizing of the process, inorganic cores always have a very low mechanical resistance when subjected to humidity for a long time.
Wet: **1h - 4h - 24h**	**Wet:** **2h - 5h**	

Temperature and humidity conditions for the ambient (25 ± 5°C and 40 ± 5% RH) and wet (25 ± 5°C and 80 ± 5% RH) storage tests have been chosen to compare the effect of humidity more easily. The characterizations performed have a comparative nature between binders, so it can be considered that a binder that is more resistant to humidity, will also have better resistance when subjected to 45°C and 80%RH.

The tests at 45°C and 40% RH / 80% RH have been useful for the understanding of the thermomechanical behavior of cores, showing that the temperature alone does not have a considerable effect on the mechanical properties, while, when its combined with the humidity, the temperature effect is severe. One hypothesis is that temperature serves as a catalyst for humidity uptake reactions, as explained in section 2.3.1.

4.1.2 MASS VARIATION (HUMIDITY UPTAKE)

The results are shown in Figure 23:

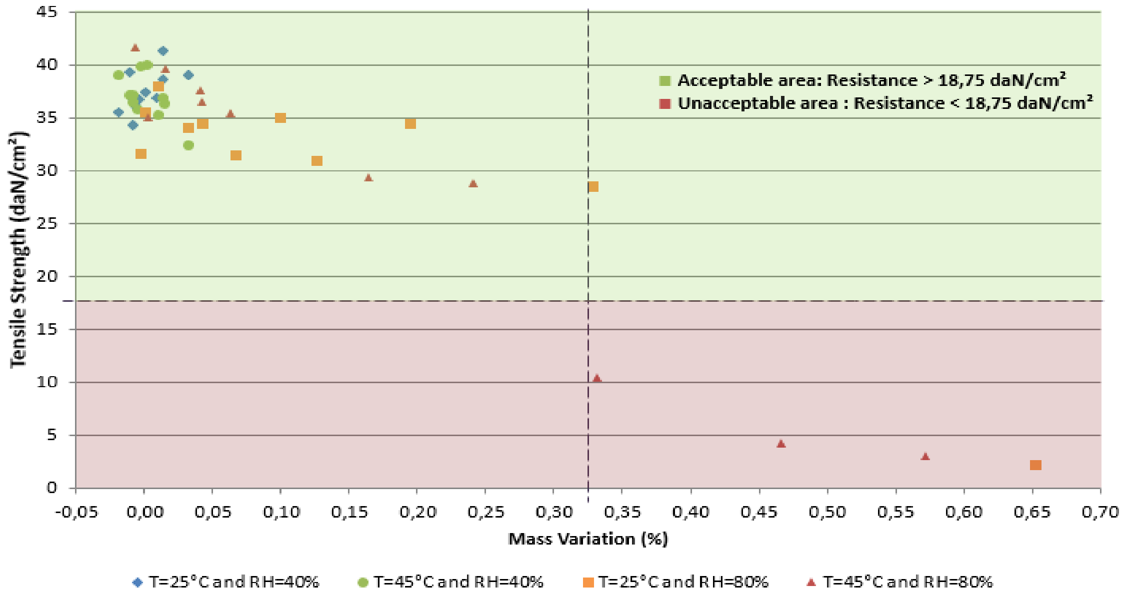


Figure 23 - Relation between the mechanical strength of cores and humidity uptake - Between 5 min and 24h

It has been concluded that, from a mass variation greater than 0.33%, cores are no longer valid concerning the mechanical strength, according to the bottom threshold of the Montupet's specifications for the humidity resistance (18.75 daN / cm²).

4.1.3 VARIATION OF GASSING AND COOKING TIMES

The results are as follows (Figure 24):

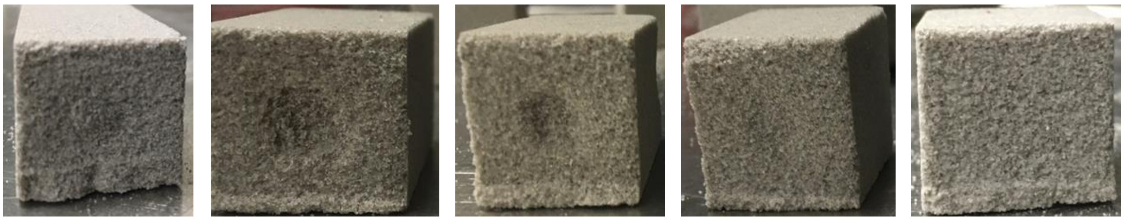


Figure 24 – Cross section of specimens subjected to different cooking and gassing times (10 - 15 - 20 - 25 - 30 seconds, respectively)

It can be noticed that, after a cooking time of 30 seconds, the specimen has been completely cooked. Hence, it is possible to state that cores are undercooked or overcooked, when cooked for less or more than 30 seconds, respectively.

Then, in order to quantitatively analyze the effect of the cooking of cores and compare with the 40 seconds test carried out previously, a new test with a cooking and gassing times of 10 seconds has been done.

The result is shown in Figure 25:

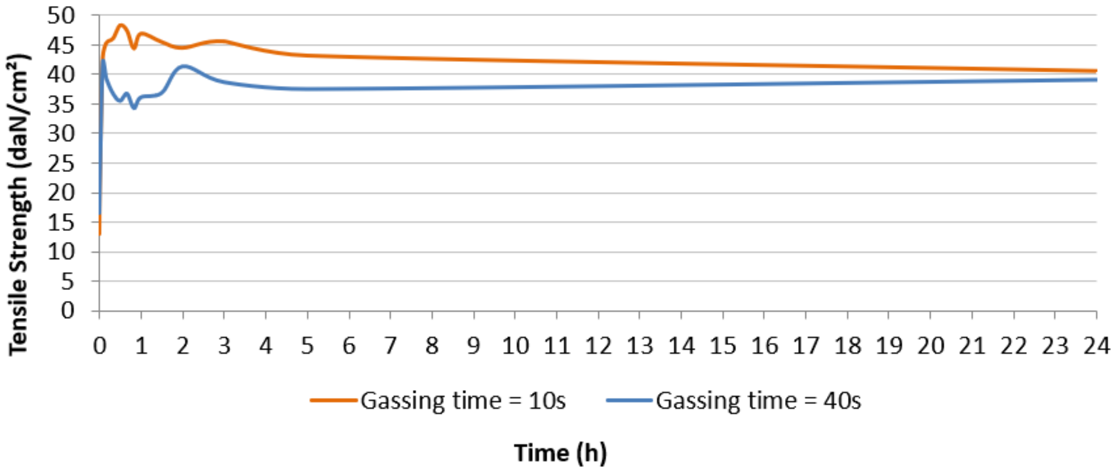


Figure 25 - Comparison of mechanical behavior of inorganic cores on different gassing times - Between 0h and 24h

It is possible to see that, except at 0h, all the mechanical strengths of the cores “cooked” for 10 seconds were above those at 40 seconds, showing that, probably, there is a loss of resistance due to the overcooking. After analyzing the fracture surface of the specimens in a SEM, it has been found that the binder bridges between sand grains are hollow for longer gassing times (Figure 26), which can explain the degradation of the cores’ mechanical properties.

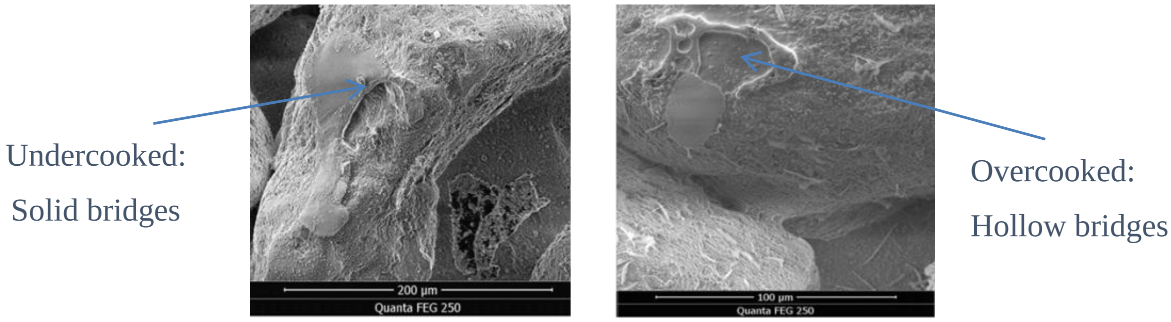


Figure 26 - SEM analysis of the fracture surface of undercooked inorganic cores (left) and overcooked cores (right)

As a result, various 3-point bending tests (CaraMécas) have been carried out for several gassing and cooking times (10, 15, 20, 30 and 40 seconds) at different storage times (0h, 2h). This time, 4 test pieces have been broken in order to have a better repeatability of the tests. The results are displayed on the following graph (Figure 27):

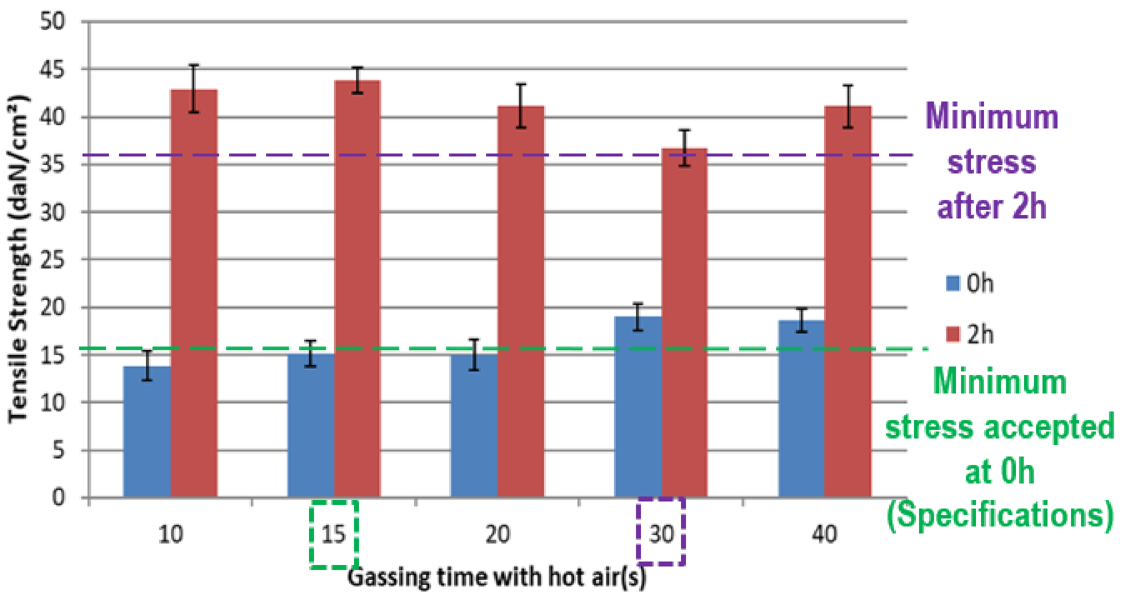


Figure 27 - Comparison of CaraMécas from cores subjected to different cooking and gassing times

After analyzing the results of these tests, 2 different cooking and gassing times have been chosen to carry out the mechanical characterizations of the inorganic cores, for the following reasons:

- **15 seconds:**
    - Lowest time to reach the minimum stress of the Montupet's specification at 0h (15 daN/cm²);
    - Time with the highest stress after 2h.
- **30 seconds:**
    - Time with the highest stress at 0h;
    - Time with the lowest stress after 2h;
    - Possibility of comparison with the supplier and competitor;
    - First time for which cooking of the core's interior is sufficient.

In addition, the two different times give the possibility to simulate two types of cooking on the production cores: thin or thick cores, willing to have a more precise characterization of the production.

4.1.4 NUMBER OF SPECIMENS (REPEATABILITY)

Figure 28 shows the results obtained by testing 2, 4 or 6 specimens for 12 conditions.

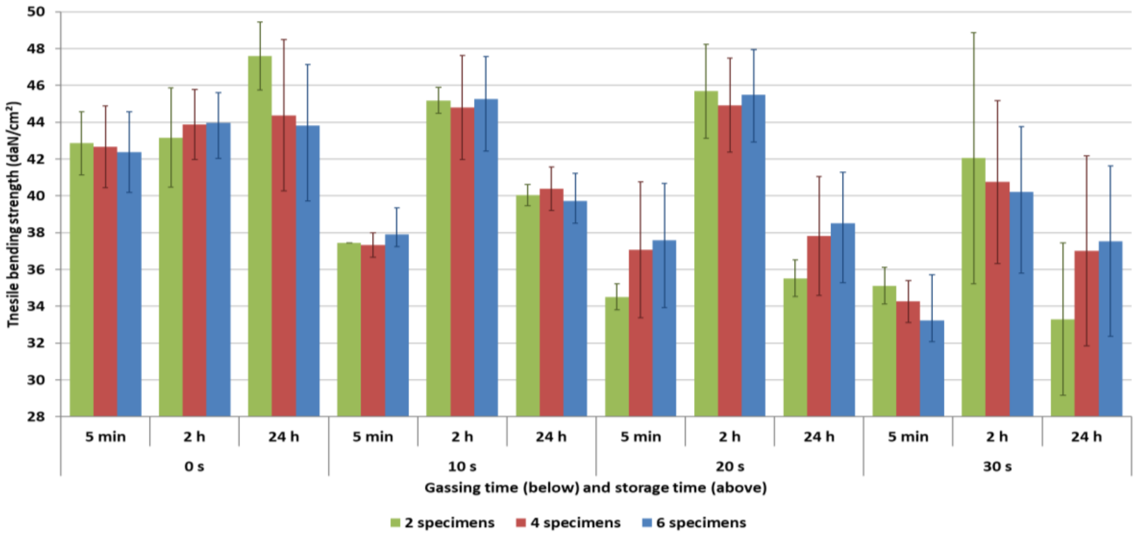


Figure 28 - Averages and standard deviations of the tests performed by simulating different repeatabilities (2, 4 and 6 specimens)

It can be concluded that the averages and standard deviations of the tests with 4 and 6 specimens were very similar to the conditions tested above. Also, their relative errors are, on average, 1.4 %. Hence, the number of specimens can be decreased per test without influencing the results. This reduction means savings in time, energy and raw materials. On the other hand, it cannot be decreased down to 2 specimens, since the averages and the standard deviations are significantly changed.

4.1.5 VARIATION OF THE GRANULOMETRY OF THE SAND

The result is shown in Figure 29 :

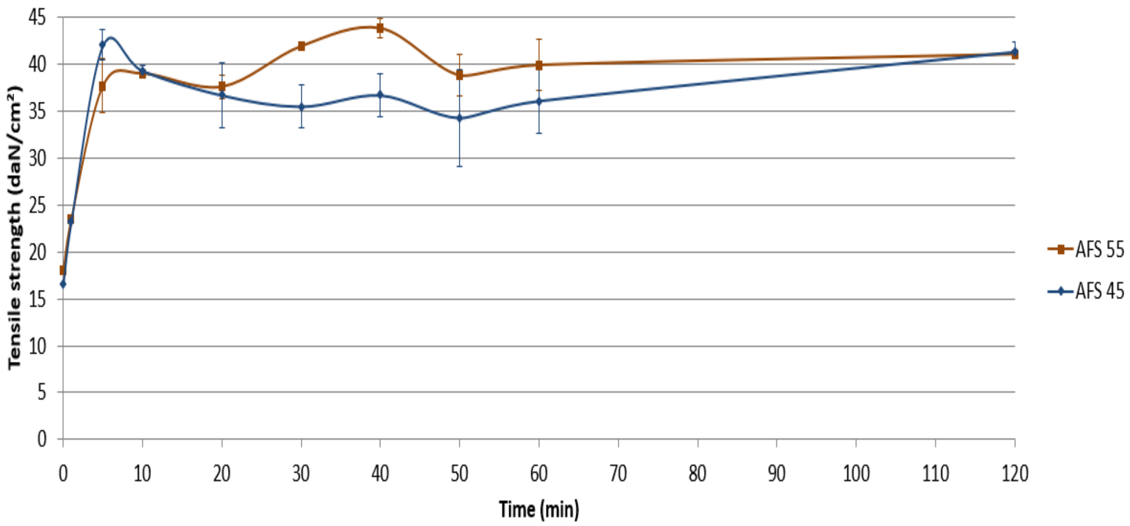


Figure 29 – Comparison of the stress of different sand AFS as a function of time

It can be noticed that the shape of the two curves and the tensile strengths are roughly similar, so it is possible to use the results with AFS 45 for the initial tests plan. Though, a probable reason why the cores made with sand AFS 55 are usually more resistant, is the greater specific surface of their grains of sand. The cores are therefore more compact and less porous, so they are more resistant.

4.2 IMPLEMENTATION OF A NEW STRAIN TEST

In this section, the most important results of the strain tests have been selected to be shown and analyzed.

First, given the repeatability of the curves (Figure 30), tests have only been repeated twice by condition, simplifying the analysis by making them much faster.

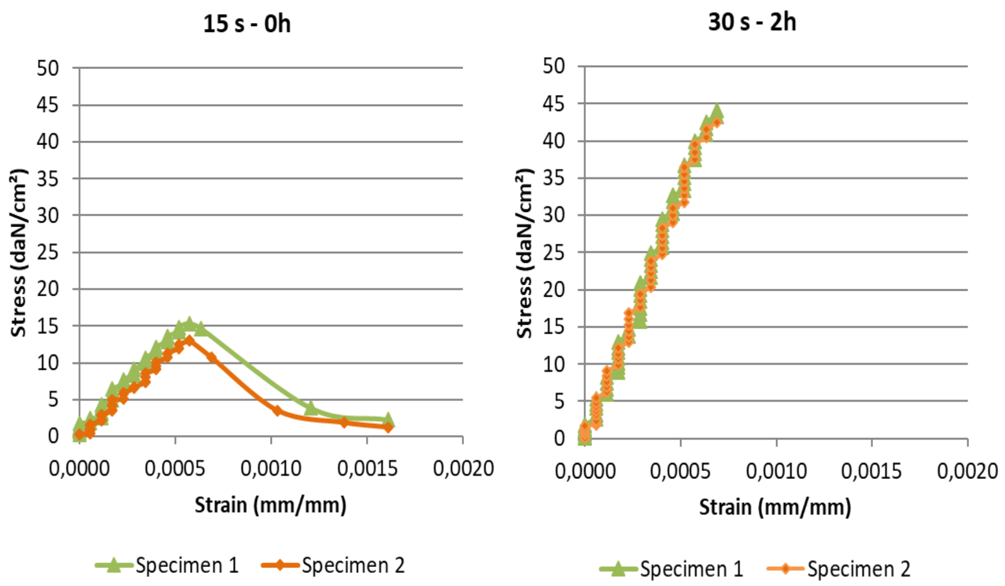


Figure 30 - Analysis of the curves' repeatability

Also, it has been decided to carry out tests under humid conditions only for the characterization of the humidity resistance, so as not to overcharge the protocol with a redundant test, even if the humidity impacts the rigidity of the cores (Figure 31).

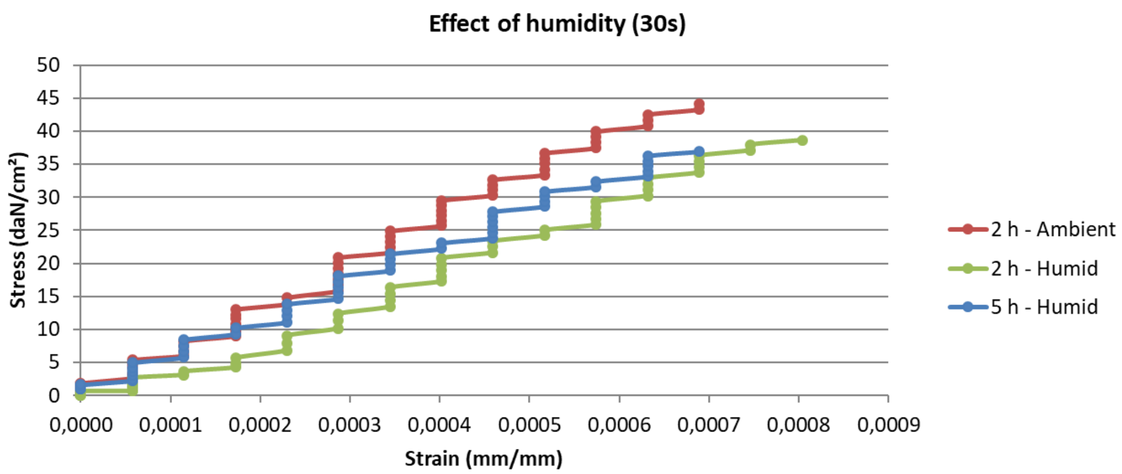


Figure 31 – Effect of humidity on the Stress x Strain curves

Additionally, by analyzing the curves below (Figure 32), it has been decided to keep a single cooking and gassing time of 30 seconds, as this was the most critical time at the maximum strain before rupture. The strains corresponding to the tensile strengths are marked in orange in the figure.

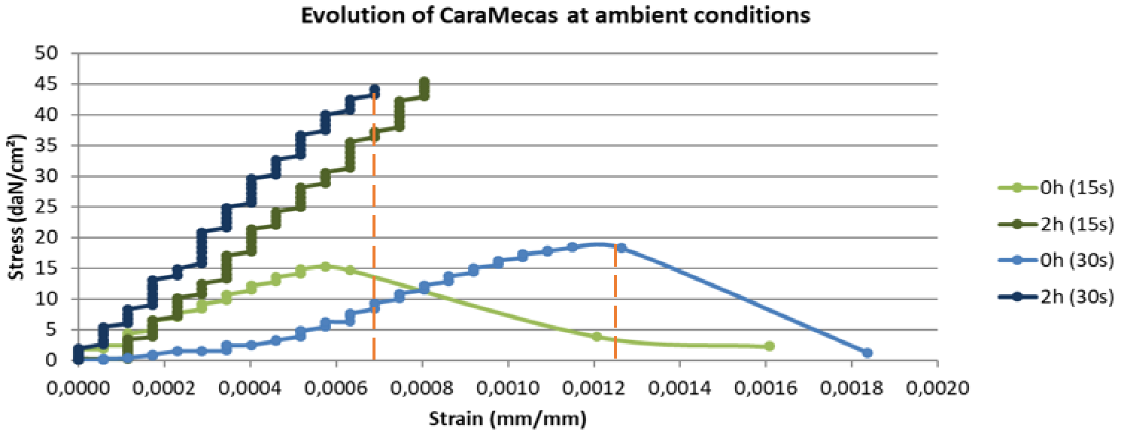


Figure 32 – Influence of cooking and gassing times on the Stress x Strain curve

Finally, for the protocol, it has been analyzed the strain at 0h, when the cores are less resistant, but more deformable, but also after 2 hours, when they are stiffened and fragilized.

4.3 ADV (ACID DEMAND VALUE) AND PH OF RECLAIMED SAND

The results of the comparison between the ADV and the pH of cores are shown in Figure 33:

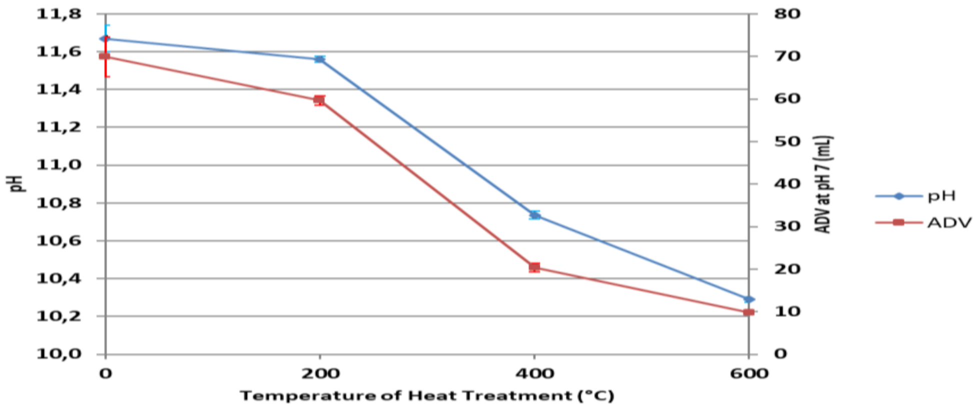


Figure 33 – Comparison between ADV and pH

By analyzing the curves above, it is possible to state that the pH and the ADV vary in the same way for reclaimed sand from inorganic cores, as the two curves have very similar behaviors. Therefore, from these results, it can be concluded that it is possible to replace the ADV test by the pH test, because the latter:

- **varies in the same way as the ADV;**
- **is faster:** about 25% of ADV time (5 min for pH against 20 min for ADV);
- **is less expensive:** no use of acid or base;
- **is more precise:** the standard deviation is smaller.

The result of the influence of ADV on core's mechanical properties is shown in Figure 34:

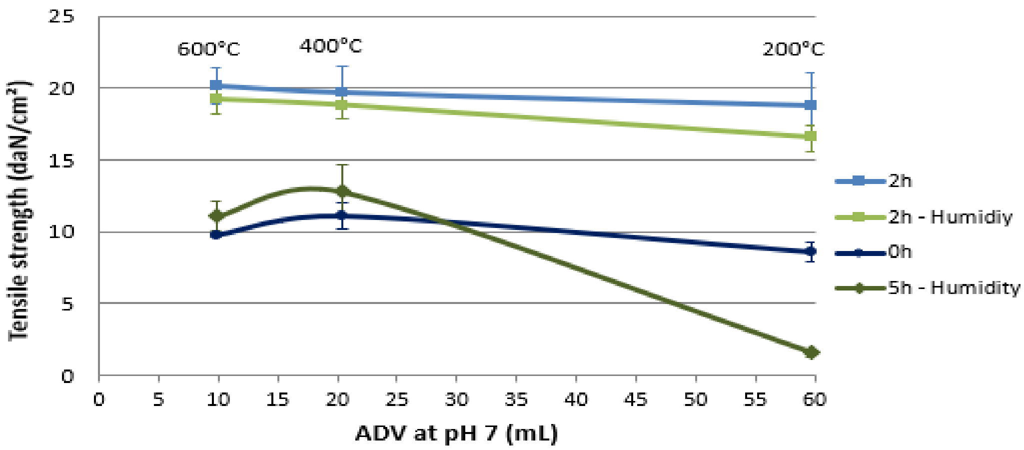


Figure 34 - Influence of the ADV on core's mechanical properties

Since the reclaimed sand has not been sieved to remove fine grains and those tests were just a small-scale representation of the highly complex real process of reclamation, it was expected that the results of the CaraMécas would be lower than the ones with 100% new sand or from the reclamation company (FATA). So, using the graph above, it can be qualitatively verified that:

- **10 < ADV < 20 ml:** a priori, it does not have a direct correlation between ADV and flexural strength;

- **ADV > 20 ml:** drop in mechanical strength, especially when the cores are exposed to humidity.

4.4 PERMEABILITY

The results are shown in Figure 35:

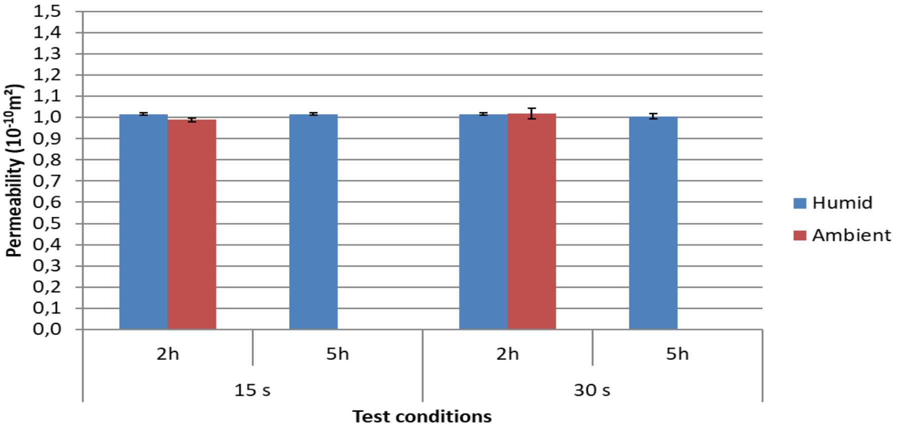


Figure 35 – Results of the Permeability tests

After analyzing the previous graph, it can be concluded that gassing, storage, and humidity conditions do not affect core's permeability. So, it has been chosen a cooking and gassing time of 30 seconds, since the permeability cylindrical specimens have a bigger diameter than the usual bending ones (used in CaraMécas and Humidity Resistance tests) and it will be more representative of a production's cooking time.

Since the standard deviations are small and with one shooting head (where is place the mix of sand and binders) it is possible to produce up to 4 cores correctly, 4 specimens per condition have been chosen, for the repeatability of the tests. After running tests on a different supplier, it has been seen that the permeability values vary according to the binder, so this is a feature that is much more dependent on the nature of the binder than the core's exposure conditions.

5. DISCUSSION OF RESULTS

Concerning the material properties of foundry cores bonded with inorganic binders, the experiments carried out have made possible to conclude that:

- Their flexural strength is greatly affected by high humidity, corresponding to a 95% drop after 24 hours of storage at 80% RH. Hence, storage humidity must be controlled, remaining below 40% RH, as used in the CaraMécas tests;
- They are very ductile after being ejected from the coring machine (when they are still hot), a factor that can cause deformation of cores and, consequently, metal parts;
- They have much higher ADV values (after reclamation), indicating potential contamination of reclaimed sand from inorganic binders, preventing their re-use in the process as "new" sand. Consequently, ADV tests on reclaimed sands must be always carried out before sending them back to the production cycle;
- Their permeability to the gases generated during casting (water), when compared to the organic cores, are lower. Which means that these gases are trapped inside the cores, causing possible internal defects of the metal parts, such as porosity or even bubbles.

Furthermore, in order to optimize the characterization tests of sand and cores used in the foundry industry, it has been possible to:

- Reduce the number of specimens from 6 to 4 per test (CaraMécas and Humidity Resistance), without losing the precision and accuracy of the measurements, which saves time, money and energy;

- Create a new strain test to obtain the stress and strain curves of the core specimens and their subsequent analysis, aiming to acquire useful information about the casting step;
- Implement two different cooking (gassing) times of the cores in the "hot box" process, allowing the mechanical characterization of thinner (well cooked) or thicker (undercooked) molds;
- Replace the ADV test for a simple pH test (faster, cheaper and more precise) for reclaimed sand, by showing that they both vary in the same way for sands reclaimed from inorganic cores.

As stated in section 3.1.1, one of the main objectives of this work was to write the characterization protocols with the modifications implemented. For each test, a different protocol has been written, explaining all the steps to be followed for repeatability. As a result, the proper functioning of the characterization process has been guaranteed, regardless of the operator. Furthermore, a total of 5 protocols have been optimized and 3 created.

Besides, the new specifications have been drafted by analyzing all the results obtained during this work and those of the Montupet’s database, with organic and inorganic binders. This analysis has been made on binders considered to be historically efficient by the company, in order to calculate the average values, of each test, as well as the percentage of tested binders that meet the specifications.

Also, an entire new database has been created, in order to improve the comparison between cores bonded with inorganic and organic binders.

6. CONCLUSIONS

The main conclusions concerning the material properties of foundry cores bonded with inorganic binders, that have been obtained throughout this work, are:

- ↑ Humidity (80% RH) → ↓ 95% of their flexural strength after 24 hours: controlled storage (below 40% RH) is needed
- ↑ Ductility after being manufactured (when still hot): possible deformation of cores and, consequently, metal parts;
- ↑ ADV values → potential contamination of reclaimed sand from inorganic binders: tests must be always carried out on reclaimed sands;
- ↓ Permeability → gases during casting can be trapped inside the cores: possible internal defects of the metal parts, such as porosity or bubbles.

Concerning the optimization of the characterization tests of sand and cores used in the foundry industry, through this work, it has been possible to:

- ↓ Number of specimens per test, conserving precision and accuracy of measurements: saving time, money and energy;
- Create a new strain test → obtaining stress and strain curves for core specimens: acquiring useful information about the casting and manipulation steps;
- Replace the ADV test for the pH test: gaining time, money and precision.

Finally, through this work, critical characteristics of inorganic cores have been detected, as well as several improvements in their characterization process, in order to adapt them to these new binders. Therefore, foundry companies are now able to implement a more respectful process, integrating into a world that increasingly considers the quality of the environment and employees' health as a priority, by the implement of legislations that block the use of organic binders in the foundry industry.

7. PROPOSAL FOR FUTURE WORK

This project offers perspectives that could not be implemented during the project period:

- **Improvement of the apparatus of the strain test:**
    - Since it has only been used equipment (acquisition center and power supplies) already available in the laboratory, they are not optimized for this test. The acquisition dates from the 90s and it was necessary to connect all the equipment and the sensors with several electric cables. It would be necessary to buy newer equipment that assembles the functions of the different power supplies and the power plant, for reasons of operation and security;
    - As the hull-knife of the force sensor needed tape to reduce the backlash, it will be necessary to redesign this device, considering the shape of the sensor.
- **pH analysis of reclaimed sand from FATA company:**
    - Studies need to be continued concerning the means of control of the reclamation of the sand used in inorganic cores. In particular, to set the acceptable values of ADV and conductivity for reclaimed sands.
- **Implementation of a new decoring test:**
    - The test currently used to assess the decoring is fairly simplistic and does not represent the complexity of the process (it is rather a friability test on furnace cores treated at 450°C). The investment in a "mini-decoring machine" would allow tests to be more representative of the production castings.

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