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# DETERMINATION OF THE MECHANICAL PROPERTIES OF CEMENTITIOUS MIXTURES WITH SEDIMENTS: EXPERIMENTAL AND NUMERICAL APPROACHES

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## ABSTRACT

Sediment management is increasingly oriented towards valorization processes in several fields. Many researches have been conducted on the characterization of sediments and the possibility to be used as raw materials in the field of civil engineering. The overall objective of this contribution is to set-up a numerical modeling strategy with a validation on the basis of experimental results. The mechanical strengths (flexural and compressive) and the elastic modulus were investigated. Five mortars were formulated with different substitution of sand by sediments (from 0% to 40%). A numerical model was built using the finite element method (FEM) based on homogenization technique. This model allows the prediction of the global mechanical behavior of the mortars based on the knowledge of the mechanical behavior of each phase constituting these samples. The model was validated through an example of a microstructure of a BOF (Basic Oxygen Furnace) slag from literature work. The microstructure has been observed using Scanning Electron Microscope SEM in order to identify the major phases in presence. Besides, the elastic properties of these phases have been determined using an instrumented indentation technique coupled with SEM. The obtained results show a good agreement between numerical and experimental. The model is validated and can then be applied on the mortars. The mechanical properties of the formulated mortars decrease with the increase of sediment substitution similarly to the obtained results in literature.

*Keywords: Dredged sediments, Mechanical properties, SEM, Indentation, Homogenization.*

## INTRODUCTION

The accumulation of sedimentary particles at the bottom and on the edges of watercourses and shipping lanes as well as in seaports leads to their clutter. For navigable waterways, it slows the flow and prevents activities of transportation. In order to maintain and restore these accesses, it is necessary to dredge or clean up regularly from coast and estuarine lines throughout the world. Currently, the French, European and international legislation aim to preserve the environment. In this context, due to the shortages of natural resources and the sustainable development approach adopted by several countries, sediment management is increasingly oriented towards valorization processes by respecting technical, environmental and economic criteria. Many studies on the reuse of sediments in mortar or concrete were investigated [1-4]. Some authors investigated the possibility of using sediments as replacement of sand in the cement-based materials while others as a replacement of cement.

All the previous researches on the valorization of sediments lies on experimental work. High number of tests should be performed in order to assess the influence of sediments on the formulated materials.

The aim of this study is to provide a new approach for valorization based on a numerical model. This approach is very challenging due to the different techniques needed for the developed model. Besides an experimental approach is conducted on five formulations of mortars with different substitution percentages of sand by uncontaminated marine sediments.

## MATERIALS AND EXPERIMENTS

### Cement

For the formulation of mortars, a Portland cement CEM I 52.5N has been used. It is composed of over 95% of clinker and less than 5% of secondary components. It is a rapid hardening cement for early strength or higher strength class. It has a density of 3.14 g/cm$^3$.

### Sand

CEN siliceous sand (according to EN 196-1), with rounded grains was used (Class 0/5). This sand has a density of 2.6g/cm$^3$.

## Sediments

The used marine sediments were dredged in the port of Dunkirk (GPMD) located in the North of France. Leaching tests of the raw sediments were conducted in accordance with the European standard EN 12457-2 (2002). These tests allow the determination of the proportions of heavy metals and pollutants leached. The results are shown in Table 1. The values obtained are compared with the inert waste reference thresholds ISDI (Decree of October 28, 2010 concerning inert waste storage facilities). They are also served to evaluate the degree of sediment's pollution. For the used sediments, the determined values are below the threshold values with the exception of antimony (Sb) and molybdenum (Mo). Thus, the studied raw sediments are categorized as nonhazardous waste according to the French legislation.

Table 1 Contaminant percentage in raw sediments (mg/kg)

| Element symbol | Element name | Percentage (%) | ISDI limits |
|---|---|---|---|
| Sb | Antimony | 0.066 | 0.06 |
| As | Arsenic | <0.08 | 0.5 |
| Ba | Barium | 0.370 | 20 |
| Cd | Cadmium | <0.008 | 0.04 |
| Cr | Chromium | <0.03 | 0.5 |
| Cu | Copper | 0.192 | 2 |
| Hg | Mercury | 0.004 | 0.01 |
| Pb | Lead | <0.03 | 0.5 |
| Mo | Molybdenum | 0.618 | 0.5 |
| Ni | Nickel | <0.03 | 0.4 |
| Se | Selenium | <0.07 | 0.1 |
| Zn | Zinc | <0.06 | 4 |

## Experiments

### *Formulation*

Five mortars were formulated for which the siliceous sand being replaced by the same volume of sediment at replacement percentage of 0%, 5%, 15%, 30% and 40%. In the following, the mortars will be referred to MT, MT5, MT15, MT30 and MT40 respectively. Table 2 presents the composition of the various formulations investigated in 1L of volume. Note that the sediments were pre-saturated before any mixing. The effective water-to-cement (W/C) ratio was kept constant (0.49) for all formulations. These formulations were prepared according to the mixture procedure described in the European standard EN 196-1. For each mixture, three prismatic samples of 4×4×16cm$^3$ were prepared and cured until 28 days.

Table 2 Composition of the mortars (in 1L)

| Mortar | MT | MT5 | MT15 | MT30 | MT40 |
|---|---|---|---|---|---|
| Cement (g) | 507 | 507 | 507 | 507 | 507 |
| Sand (g) | 1526 | 1450 | 1298 | 1068 | 916 |
| Water (g) | 249 | 249 | 249 | 249 | 249 |
| Sediment (g) | 0 | 95 | 285 | 570 | 760 |
| W/C | 0.49 | 0.49 | 0.49 | 0.49 | 0.49 |

### *Mechanical strength*

The mechanical strength of the prismatic mortar samples (4×4×16cm$^3$) were determined in accordance with the European standard EN 196-1. Flexural and compressive tests were carried out using a mechanical INSTRON testing machine. These tests were performed on the sediment-based mortars after being cured in water for 28 days. Results are the averages of three samples.

### *Elastic modulus*

A method has been developed to determine the elastic modulus of the sediment-based mortars. It consists in recording the deformation of the tested specimen when subjected to a compression imposed by the INSTRON testing machine (with a displacement control). The elastic modulus E (MPa) is obtained from the slope of the elastic part of the stress–strain curve. The deformation was measured using a strain gage (SG) installed at one side of the tested specimen using an adequate glue (X60). The length of the strain gage as well as the displacement rate imposed by the compression machine were optimized in order to determine an accurate value of the elastic modulus.

Note that in order to compensate the effect of the temperature on the deformation result, a dummy gage (identical to the active strain gage) was installed on an unstrained sample of the same material as the tested specimen. In this way, the deformation caused by the temperature change in the dummy gage can be calculated and then subtracted to that given by the active gage to obtain at the end the real representative deformation of the material. The elastic modulus results are the averages of three measurements.

## NUMERICAL MODEL

The mechanical behavior of the sediment-based mortars can be predicted numerically, avoiding the complicated and costly experiments. The mortar is a complex material with a variety of heterogeneities. The determination of the macroscopic response of such heterogeneous materials is of a great interest in engineering applications and large number of researches have been made in the last years [5]. The method consists in determining the macroscopic elastic behavior through the knowledge of the mechanical behavior of each phase that constitutes the tested sample. A numerical model is built using the finite element method (FEM). This model is based on the homogenization technique (theory of effective properties), by which the heterogeneous material is replaced by an equivalent homogeneous continuum [6]. The latter should respond globally to any load in the same way as the heterogeneous original material. This technique is applied on a statistically representative sample of material, referred to as a representative volume element (RVE) [7], [8]. The main objective of this numerical homogenization is to predict the effective properties on the RVE. This approach has not been yet performed on sediment-based mortars.

The microstructure of the mortar sample is obtained using a Scanning Electron Microscope (SEM) coupled with an Energy Detector Spectrometer (EDS). The major phases constituting the heterogeneous mortar can then be identified. The inputs of the finite element FE model are the elastic properties of each phase: elastic modulus $E_i$ and Poisson's ratio $\nu_i$. The Poisson's ratio was assumed as 0.2. The elastic modulus $E_i$ of each phase can be determined by means of a coupled system combining a Scanning Electron Microscope (SEM) and an instrumented indentation equipment [9], [10]. This step is the key to the developed model. The main advantage of this system is the capability of performing indentation tests in a specific area (a particular phase). A pre-treatment of the image representing the microstructure of the studied sample is necessary. The microstructure was segmented into scales of grey color using conventional image analysis/processing. Filters were used to eliminate the noise produced by the microscope. FE meshing was then performed using multi-phase element method. [11]. This method consists of superimposing a regular FE mesh on each microstructure image. The obtained meshes were then introduced in the computation code. The image of the microstructure was used to attribute at each integration point, the mechanical behavior associated to the phases. Different density of meshes were applied to study the mesh sensitivity on the results. The mesh density is defined as the number of FE required to mesh the volume of heterogeneous material. For numerical simulations, a simple uniaxial compression test has been applied, defining the boundary conditions. A displacement is applied to one face of the microstructure while the opposite one is fixed. All the other faces are free. These conditions can be illustrated in Eq. (1):

$$\begin{aligned} &\overline{u}\{\text{face}(y=0,x)=0\}\\ &\overline{v}\{\text{face}(y=0,x)=0\}\\ &\overline{v}\{\text{face}(y=L,x)=d\} \end{aligned} \qquad (1)$$

where $\overline{u}$ and $\overline{v}$ are the applied displacements in x and y-directions, L is the numerical model length, and d is the imposed displacement in y-direction (Fig. 1).

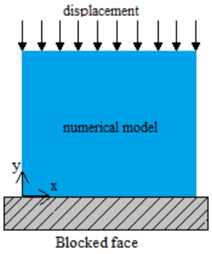

Fig. 1 Boundary conditions for the numerical approach

The homogenized effective elastic modulus can then be determined. The algorithm of the numerical model is presented in Fig. 2.

## RESULTS AND DISCUSSION

### Experimental

The flexural and compressive strengths of the sediment-based mortars are presented in Fig. 3 and Fig. 4. Average values obtained by three measurements for flexural strength and six measurements for compressive strength are shown with the corresponding standard deviation. Both the flexural and compressive strength of mortars decreased with the proportion increase of sediment.

This result highlights the influence of sediments on the mechanical behavior of cemented-materials. The mechanical strength comes foremost from the formation of cemented products. The use of marine dredged sediments as aggregates, compared to sand, may alter the hydrating processes. Organic matter which is contained in sediments are especially known to interfere with the hydration process of cement [12]. The marine sediments contain also chloride salts. Their high salinity has a negative effect on the mechanical strength development of cement-based materials [13]. Besides, traces of metals are presented in these sediments (Table 1). All of them even with small concentrations can influence the quality of the obtained mortars. They might affect adversely the mechanical strength by changing the mortar's average porosity.

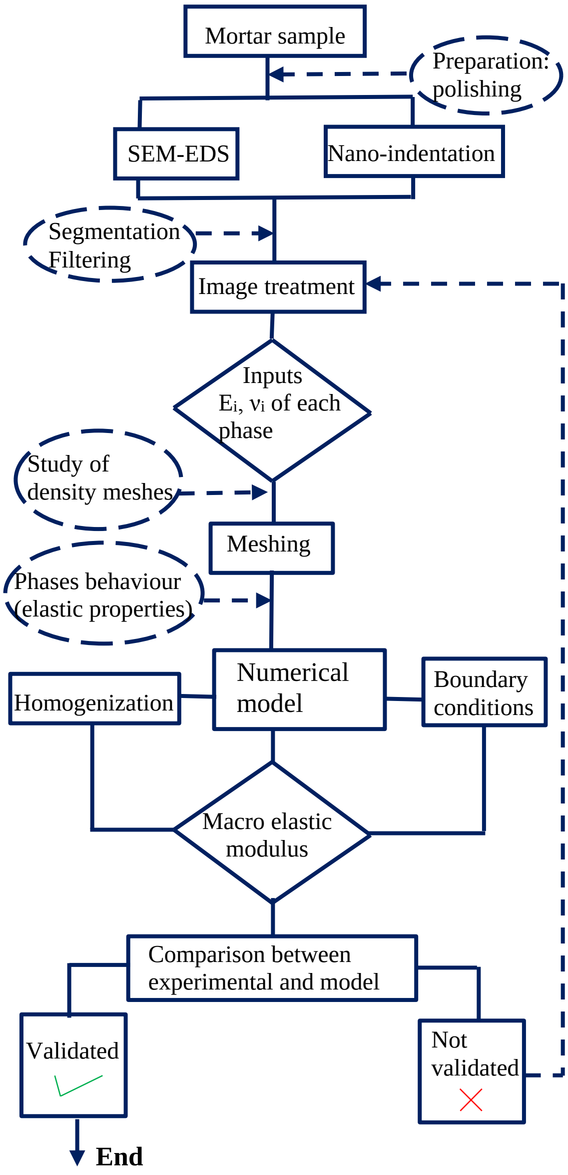

Fig. 2 Algorithm of the numerical model

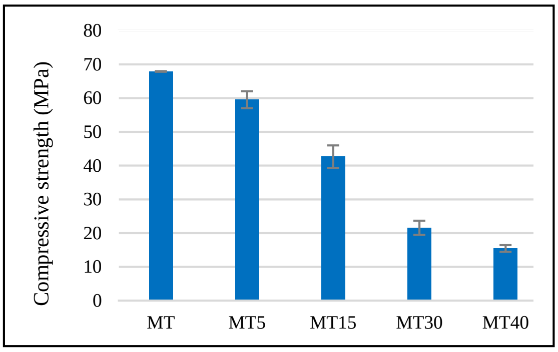

Fig. 3 Compressive strength as function of mortars

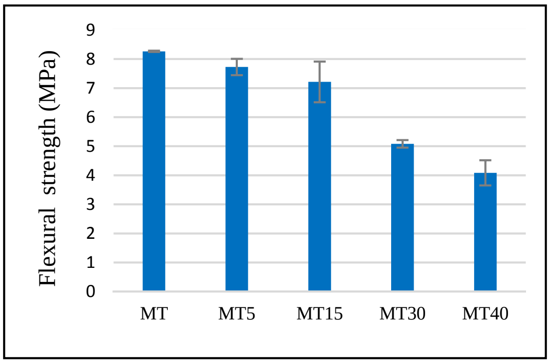

Fig. 4 Flexural strength as function of mortars

The influence of sediments can be refined by presenting the percentage of the compressive strength of sediment-based mortars with respect to the unsubstituted reference mortar (MT) as a function of sediment percentage (Fig. 5). The compressive strengths of mortars decrease linearly with the amount of substitution of sand by sediment up to 30% of replacement. Between 30% and 40%, the decrease is barely noticed. For MT5, the compressive strength attains 87% with respect to MT while 62% for MT15, 31% for MT30 and 28%. For MT40. Substituting more than 30% of sand by sediment has no additional effect on the mechanical behaviour of mortars. This result can be explained by the repartition of particles in the microstructure of the cemented-materials.

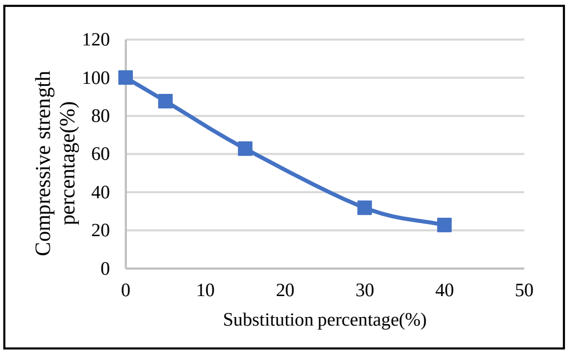

Fig. 5 Compressive strength percentage as function of the amount of sand substituted by sediment

The average elastic modulus and the standard deviation of three measurements for each mixture of sediment-based mortars are presented in Table 3. The errors on the measurements are less than 5%. The percentage of the elastic modulus of mortars with respect to the unsubstituted reference mortar (MT) is also shown. The elastic modulus E decreases when the percentage of replacement of sand by sediment increase. The variation of E with respect to MT is s similar to the results obtained in Fig. 5. The Young modulus decreases approximately by 80% when substituting 40% of sand by sediments.

The Young modulus depends heavily on the modulus of the aggregates that compose the cemented-materials as well as on their form and texture [14]. This decrease with the sediment substitution can be explained by two reasons. In general, sediments have high porosity which generate an increase of the mortars porosity and thus the diminution of the elastic modulus. Besides, sediments have low resistance compared to natural aggregates. Substituting sand by sediments leads to low values of the young modulus.

Table 3 Elastic modulus of mortars

| Mortar | MT | MT5 | MT15 | MT30 | MT40 |
|---|---|---|---|---|---|
| E (MPa) | 35076 | 31554 | 22710 | 12417 | 7678 |
| Standard deviation (MPa) | 1524 | 1049 | 2125 | 396 | 229 |
| E Percentage | 100 | 90 | 65 | 35 | 22 |

## Numerical

The microstructures images of the sediments-based mortars were not performed yet. Thus, the numerical approach was validated through an example of a microstructure of a BOF (Basic Oxygen Furnace) slag studied experimentally by D. Betrancourt et al [10]. Five phases constituting the slag were identified (Fig. 6) with the corresponding proportion and elastic modulus $E_i$ obtained from the nano-indentation technique. Table 4 resumes the different phases characteristics.

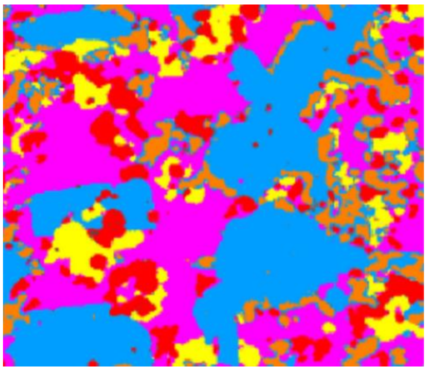

Fig. 6 Phase identification in the slag [10]

Table 4 Characteristics of slag's phases [10]

| Phase | Compound | Area | E (GPa) |
|---|---|---|---|
| Phase 1 (pink) | $C_2F$ | 30.5 | 127±22 |
| Phase2 (orange) | $C_2S$ | 10.1 | 118±6 |
| Phase 3 (blue) | $C_2S+C$ | 35.5 | 106±4 |
| Phase 4 (yellow) | C | 9.2 | 67±8 |
| Phase 5 (red) | FeMgMn | 14.7 | 139±14 |

The treatment of the image was performed (segmentation, filtering) and six different densities of meshes were studied (from 2500 to 250000 elements). FE meshing was performed using the multi-phase element method (Fig. 7).

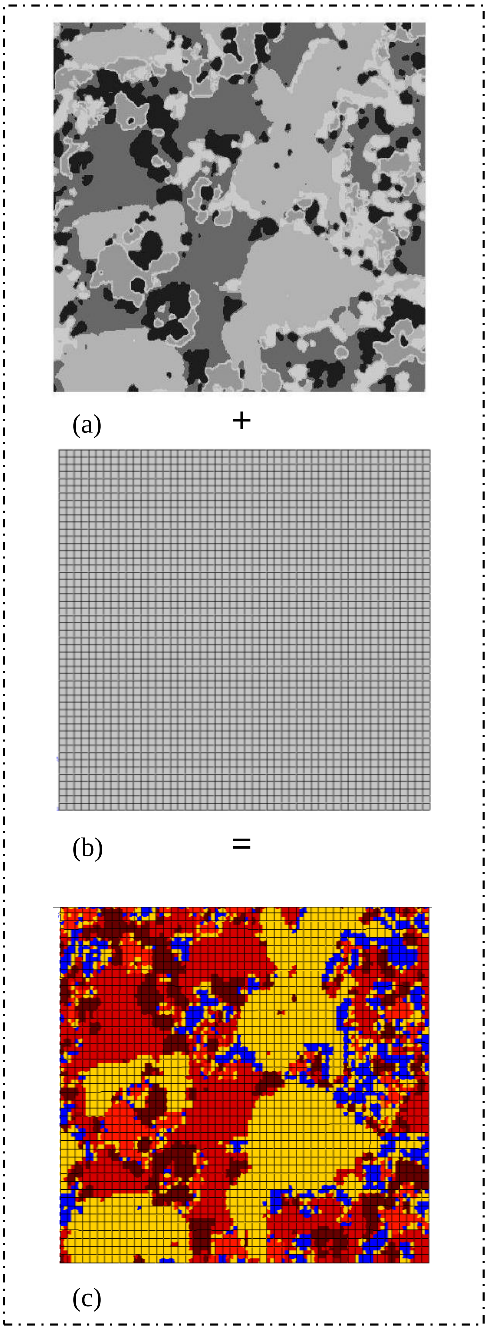

Fig. 7: Multi-phase element method: (a) Microstructure image after treatment process, (b) 2D FE-grid (one mesh type) and (c) Mesh of microstructure (colors here only for illustration purpose)

The homogenized elastic property of the material was then determined after numerical computations. A value of E= 109 GPa was obtained regardless the mesh density. The sensibility of the mesh is considered negligible on the macroscopic result. This value is in conformity of what has been obtained by D. Betrancourt et al. [10] who have estimated the global elastic property by the application of mixtures law (Table 5).

Table 5 Estimation of global elastic modulus by mixture law models, from the estimated values in Table 4

| | Voigt | Reuss | Voigt-Reuss-Hill |
|---|---|---|---|
| E (GPa) | 115 ± 12 | 111 ± 11 | 113 |

The numerical approach was validated. The influence of each phase on the global property can be studied. Additional neglected phases can be introduced on the microstructure as well, such as pores phase, to optimize the results.

This approach can be applied on 3D images which can be obtained by performing X-ray computed Tomography (XCT).

## CONCLUSIONS

The present study aimed to minimize the tests required for evaluating the mechanical behavior of sediment-based mortars. Five formulations of mortars were prepared with different substitution percentages of sand by uncontaminated marine sediments. Compressive and flexural tests were performed on the formulated mortars. The elastic modulus was also investigated using specific strain gages.

A numerical model was developed to assess the global mechanical property of the mortars. This model was built using the finite element method based on the homogenization technique. The main key for this approach is the knowledge of the elastic property of each phase constituting the sample in study. For this purpose, an instrumented indentation technique coupled with SEM should be performed.

The results show that the flexural and compressive strengths of mortars decrease when the substitution of sediment increases. This decrease is linear up to 30% of replacement. The elastic modulus decreases as well with the percentage of substitution of sand by sediment. This property depends mainly on the modulus of the aggregates composing the cemented-materials. The numerical model was validated on a slag microstructure. The macroscopic elastic property was in conformity with the value obtained experimentally by the authors of the mentioned work. This approach was not applied on the mortars. It will be the object of another detailed study.

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