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Characterization and valorization of dam sediment as ceramic materials

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Abstract*— the present work aims at characterizing and promoting the use of two fine-grained sediments (referenced ISE and RHI) from hydroelectric reservoirs of two areas located in France. Due to their chemical and mineralogical compositions, these sediments could be used for the production of ceramic-based construction materials, like bricks and tiles. The materials obtained by extrusion were fired at different temperatures. Their structural and thermal transformations upon firing were investigated in connection with the properties of use, such as water absorption, bending strength, and thermal conductivity. Results show that physical and mechanical properties developed by the sediment-based materials are close to those of conventional fired clay bricks. ISE-based samples exhibit appropriate characteristics, such a bending strength of 7.5 MPa and a thermal conductivity value of 0.45 $W.m^{-1}.K^{-1}$ without significant composition modification after firing at 880°C. RHI sediment requires addition of a plastic body (RR40® clay) to achieve satisfactorily plasticity regarding the extrusion process. The addition of 10 mass% and 20 mass% of RR40® clay in the RHI sediment improves the fired products properties. The bending strength increases from 7.5 MPa to 8.7 MPa and water absorption is decreased from 28% to 20% when adding 20 mass% of RR40® clay and firing at 880°C. Furthermore, the RHI sediment with 20 mass% of added clay, after firing at 1095°C, may also match potential use as majolica-like tiles according to literature.

Index Terms— **dam sediments, ceramic materials, waste management, thermal transformations.**

I. INTRODUCTION

Sediment is a natural material which accumulates in harbors, canals, rivers lagoons and reservoirs. It is a relatively heterogeneous product and exhibits a broad composition distribution, combined with the presence of high amount of water and organic matter [1-5].

For a given site, the nature and volume of deposits depend on different factors such as catchment areas, soil geology, climate regime of the valley, river's hydrological regime, catchment area vegetation cover type or level of human activities i.e. farming practices, urban sanitation, industries, etc. The management of dredged sediments has become a major concern for all ports, waterway authorities, and more recently hydroelectric plants and dam operators. Currently, more and more regulations are imposed and usual practices are liable to be called into question.

The silting is an inevitable phenomenon appearing in dams with a variable importance depending on location and sediment management options. For hydraulic and efficiency criteria of the structure or to use the control the water use, it is important to manage these sedimentary deposits. Requirements related to the operation and the environment may force operators to deviate from the conventional solutions of management that consist in reintegrating sediments in watercourses. If dredged materials i.e. sediments, are stored or selected for land management, they enter into the European List of Waste. However, the dredged sediment might be considered as a natural and renewable resource. Therefore it is a challenging task for managers to find beneficial uses for these materials.

Several industrial sectors where sediments could be used have been identified, among them: erosion control, aquaculture, agriculture, forestry, manufactured topsoil [6], alternative raw material for Portland clinker production [7, 8], infrastructure works including sub-base material for road construction [6, 7, 9-11], manufacture of light-weight aggregates [12-14] and as mineral addition in concrete matrices [9, 10, 15, 16]. The valorization of these wastes as secondary raw materials in the production of construction materials could hinder the depletion of natural resource and solve the disposal of industrial wastes. Among construction materials, traditional clay-based materials are heterogeneous products that may accommodate different inorganic wastes or sub-products without a significant modification of the production process or the final properties [11, 17, 18]. Consequently, the incorporation of industrial wastes or sub-products in bricks and tiles is contemplated in ceramic processes.

The valorization of these materials fits into the overall framework of sustainable development [19].

Because of their high content of clay minerals, the incorporation of sediments in the sector of traditional ceramics (tile, clay brick) is a very promising solution

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towards sustainable management. In recent decades, brick making has been assessed using river [12, 14, 20] and marine sediments [15, 16, 20-23], sediments from lakes and dams [13, 24, 25], and sewages [26]. The objective of these studies was to characterize the selected sediments and to investigate the appropriate conditions for their valorization in the field of traditional ceramics.

II. MATERIALS AND METHODS

A. Methods

The particle size distribution of sediments was determined using a laser particle size analyzer Mastersizer 2000 (Malvern). 2 g of sediment samples were dispersed in 30 mL distilled water containing of sodium hexametaphosphate (0.1 wt%). Prior to measurement, the suspension was desagglomerated for 5 minutes using an ultrasound source.

The chemical composition of sediments was determined using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). Prior to analysis, dried sample powders were dissolved in water using microwaves under acidic and high pressure conditions. A CEM MARS 5 microwave was used and the dissolution was achieved after a 45 min-cycle including a maximum temperature and pressure of 180°C and 3 MPa, respectively.

The specific surface area of the sediments was determined by the Brunauer Emmett and Teller method, using a TriStar II device from Micromeritics [27]. Measurements were carried out after a 16 hours degassing step at 150°C, on dried samples previously crushed and sieved to 125 µm.

The density of powders was determined using an automatic helium pycnometer of type Micromeritics Accupyc 1330 (Norcross, USA).

Prior to all analyses, the as-received sediments samples were dried at 40°C, crushed and sieved at 125 µm, and kept at 40°C to prevent rehydration.

The nature of crystalline phases of the different samples was characterized using X-ray diffraction (XRD). XRD diagrams were obtained on powdered samples with a Bruker-AXS D5000 powder diffractometer using K1 radiation of Cu and a graphite back-monochromator. XRD experiments were achieved in step-scan mode from 3° to 60° (2θ) with a counting time of 10.1 per 0.02° step.

The thermal behaviors of the raw materials were carried using a SETSYS 2400 DTA-TGA equipment (SETARAM) in the temperature range 30°C-1200°C and under dried-air atmosphere. Alumina powder, previously at 1500°C for 1 hour, was used as reference material. All analyses were performed using 60 mg of sample and reference.

Small blocks of about 6 mm x 6 mm x 10 mm prismatic samples were cut off in the samples extruded. After drying at 40°C for 48 hours, the densification behavior of these samples during a heating at 5 ◦C/min under air, was characterized using a vertical dilatometer SETSYS TMA Evolution (SETARAM).

The observation of some sintered samples microstructure was performed using a Cambridge Stereoscan 260 SEM equipped with a PGT Prism energy dispersive spectrometer. SEM specimens were polished using 12 µm, 5 µm and 1 µm diamond pastes after grinding with sandpaper and water. Prior to observation, the polished surfaces were carbon coated.

Samples have been mechanically tested using a LLYOD EZ20 device, equipped with compressive, tensile and bending test units. The deflection rate is maintained at 0.5 mm/min. The three points bending tests are carried out on 40 mm x 14 mm x 16 mm samples, the values of the flexural strength presented here are mean values over five specimens measured under the same conditions.

The thermal conductivity was determined by the hot disk method. This technique gives directly access to the thermal conductivity of materials. The measurement principle consists in inserting a probe between two blocks of material. The probe acts both as a source of heat flux and as temperature sensor. The measurement consists in imposing a voltage to the sample and in following the rise of the temperature of probe over time. The analysis of the temperature increase over time allows calculating the thermal conductivity value.

B. Materials

Fine sediment samples were collected from two French dam reservoirs operated by the EDF group for hydroelectricity production. Reservoirs are located in different regions and may constitute mineral deposits for industries. A barge and a grab were used to extract sediments. Samples of sediments were collected in hermetically sealed plastic barrels. Sediments were referenced with three letters according to their origin: ISE and RHI.

- ISE sample has a homogeneous appearance and its color ranges from brown to gray.
- RHI sample presents a muddy appearance, and contains many shells and various plant residues. It underwent a dilution with water and was passed through a 1 mm sieve to remove larger objects.

Sediments were dried slowly at 40°C to avoid any alteration of organic matter until the mass was constant [28, 29]. All characterizations of the sediments were carried on samples crushed and sieved to 125 µm.

Brick samples were shaped using the extrusion process. Moisture content of the sediment bricks, as measured on extruded bricks, varied from 24 to 30% by weight. Extruded wet brick columns were cut into brick blocks of 14 mm x 40 mm x 16 mm and thermally processed. Brick samples were dried in an oven at 40°C during 72h, the dried specimens were then fired at different temperatures, previously identified by dilatometry analysis. It should be noted that the RHI sediment-based paste presented a bad plasticity, this has made it difficult to develop brick by extrusion. Therefore, two others RHI-based bricks were tested by adding 10 and 20 mass% of a commercial plastic raw clay labeled RR40®. The chemical and mineralogical composition of RR40® is presented in **table 1** according to the technical fact sheet provided by the supplier.

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Table 1: Chemical and mineralogical composition of RR40® clay.

	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
Chemical composition (%)	54.7	39.5	2.2	2.3	0.6	0.2	trace	trace
Mineralogical composition (%)	Kaolinite (90%), Quartz (8%), Anatase and Rutile very low content							

III. RESULTS AND DISCUSSION

A. Characterization of raw materials

The particle size distribution curves (**Fig 1**), show that ISE sediment contains finer particles than RHI ones. The different physical characteristics are listed in **table 2**. Densities of the sediments vary between 2.5 and 2.7 g.cm^{-3} and are consistent with values obtained for most raw clay materials used in the traditional ceramic industry. The BET specific surface area value obtained for ISE sediment (12.45 m^2.g^{-1}) is similar to that observed for kaolinite type clays [30, 31] while the value obtained for RHI ones is lower (6.15 m^2.g^{-1}), in agreement with the obtained particle size distributions. The high water content of RHI is due to the dilution prior to sieving at 1 mm that was required to remove coarser objects (shells and plants residues).

Table 2: Physical characteristics of sediments.

Sediment	ISE	RHI
Density (g.cm^{-3})	2.66	2.55
Specific areas (m^2.g^{-1})	12.45	6.15
Water content (%)	47.3	64.8

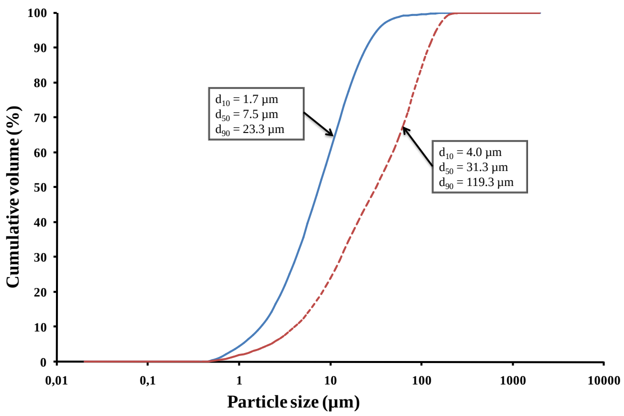

Fig 1: Particle size distribution curve for RHI (- - -) and ISE (—) sediments.

Data of the chemical composition of the raw sediments in oxide form is presented in **table 3**. It can be observed that the main oxides present in the sediments are SiO_2, CaO and Al_2O_3. Fe_2O_3, MgO, Na_2O and K_2O are also detected but in lesser amounts. The high SiO_2 content is induced by the presence of quartz particles, where Al and Si oxides can be associated with clay minerals present in the materials. The SiO_2/Al_2O_3 ratio is close to 4.1 and 6.9 for ISE and RHI sediments respectively. The latter trend suggests the presence of 2:1 clay minerals together with associated silica phases. Moreover, it is likely that RHI sediment contains more silica phases than ISE ones. A significant amount of Fe_2O_3 (6.38 wt%) in ISE may contribute to the reddish color of the fired bricks. The sediments also contained fluxing oxides, such as potassium, sodium and calcium oxides, which may promote fusion of the particles at lower temperature during the firing process. Furthermore, the great amount of CaO may indicate the presence of significant carbonates within both sediments. The MgO can be attributed to clay minerals as well as carbonates.

Table 3: Chemical composition of ISE and RHI sediments.

Oxide content (mass%)	ISE	RHI
SiO_2	**58.77**	**67.84**
Al_2O_3	**14.30**	**9.80**
CaO	**14.60**	**11.90**
Fe_2O_3	**6.38**	**3.57**
MgO	**2.07**	**3.80**
Na_2O	**1.31**	**1.24**
K_2O	**2.57**	**1.85**
SiO_2/ Al_2O_3	**4.1**	**6.9**

The XRD diagrams of ISE and RHI sediments are presented on **Fig 2**. It can be seen that the two sediments contain clinochlore ($(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$), a phyllosilicate that belongs to the chlorites group, quartz (SiO_2), calcite ($CaCO_3$), albite ($Na(AlSi_3O_8)$), goethite (FeOOH) and muscovite ($KAl_2(Si_3,Al)O_{10}(OH)_2$) as crystalline phases, whereas dolomite ($CaMg(CO_3)_2$) is also detected in RHI sediment. The crystalline phases detected using XRD are in agreement with chemical composition obtained by ICP-AES. In accordance with their chemical and mineralogical composition, these sediments are suitable to be used as raw material in a traditional ceramic [32]. In fact, most clays used for tile and brick manufacturing contain appreciable amounts of iron oxide and calcium oxide (which is usually present as $CaCO_3$ and $CaMg(CO_3)_2$). Furthermore, quartz is one of the main components in ceramic pastes.

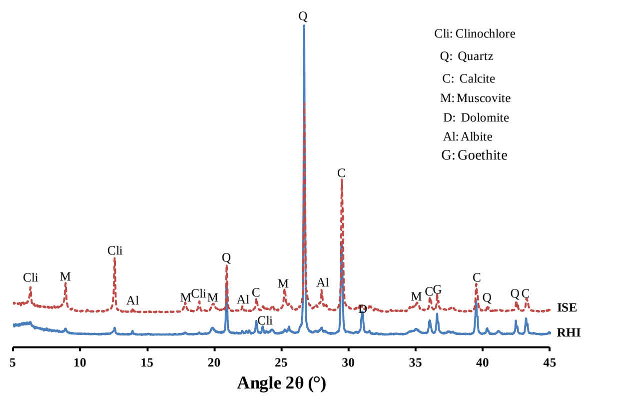

Fig 2: X-ray diffraction diagrams of sediments.

The TGA/DTA analysis results of sediments are presented on **Fig 3** (**a**) and (**b**). As seen from **Fig 3** (**a**) and (**b**), a continuous weight loss distributed between 30 and 800°C on the TG plot is found for the two sediments. The first weight loss of 0.8% and 1.5% respectively for ISE and RHI

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Volume 4, Issue 8, February 2015

sediments is observed between 30 and 200°C and is associated with an endothermic peak caused by the evaporation of the adsorbed water. All samples show an exothermic effect, with a weight loss of 0.9% and 3.1% for ISE and RHI sediments respectively in the 200–400°C interval, which are likely due to the oxidation of the organic matter adsorbed in the sediments [33]. A certain amount of organic matter is desirable since it can contribute to improve the plasticity of the green ceramic body [23, 34]. Nevertheless, a large total organic matter content may render sediments useless for brick making.

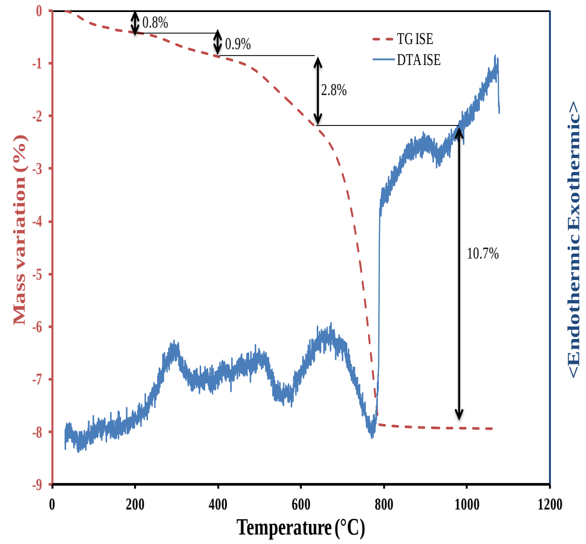
The weight loss of 2.8% and 3.2%, observed respectively for ISE and RHI sediments at 400-650°C, is attributed to the dehydroxylation of the clay minerals.

A significant weight loss of 10.7 and 12.8 % for ISE and RHI sediments respectively is associated with an endothermic peak between 650°C and 850°C. It corresponds to the decomposition of carbonates [22].

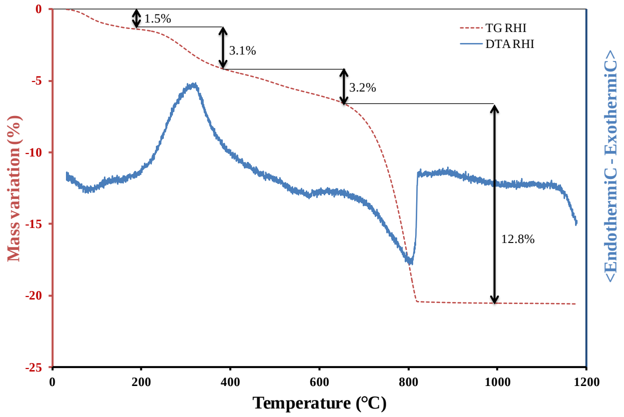
B. Characterization of the sintering behavior

The analysis of the length variations of extruded sediments sample was performed using dilatometric measurements. The obtained results are presented on **Fig 4** with a heating rate of 5°C/min and maximal temperatures of 1050°C and 1125°C for ISE and RHI samples respectively.

- For both samples, a non-linear expansion is noted from room temperature to 600°C. This behavior may include the intrinsic thermal expansion of material during heating together with additional variations due to the physical/chemical transformations related to organic matter decomposition and clay minerals dehydroxylation. The typical allotropic transformation of quartz is also observed at 575°C as expected, regarding the mineralogical composition of these sediments.



(a)



(b)

Fig 3: Thermal analysis of: (a) ISE sediment and (b) RHI sediment.

- Between 600°C and 800°C, there is a smoothing of the samples' expansions that can be linked to the relative mass losses due to the clay minerals dehydroxylation and the beginning of carbonates decarbonation (see DTA-TGA curves of Fig 3).
- From 800°C to 950°C, a first significant shrinkage is noted for ISE (0.5%) and RHI (1.4%) sediments. The observed trend is in relation with the phases structural reorganization and consolidation of the powder compacts without significant porosity change according to literature [35, 36]. Characteristic temperatures of the end of this shrinkage are 880°C and 900°C for ISE and RHI sediments respectively.
- Between 950°C and 1000°C, ISE specimen undergoes subsequent expansion that may result from the high temperature phase's formation and the evolved CO_2 through the decarbonation reaction previously mentioned. The same trend is observed for RHI specimen between 950°C and 1100°C.
- Above 1000°C and 1100°C for ISE and RHI sediments respectively, the supplementary densifying shrinkage is observed. In the case of ISE sample, the shrinkage level is similar to the first shrinkage observed at 880°C that is 0.7%. For RHI sample after the sintering at 1125°C, the shrinkage amplitude is very high, close to 6.1%.

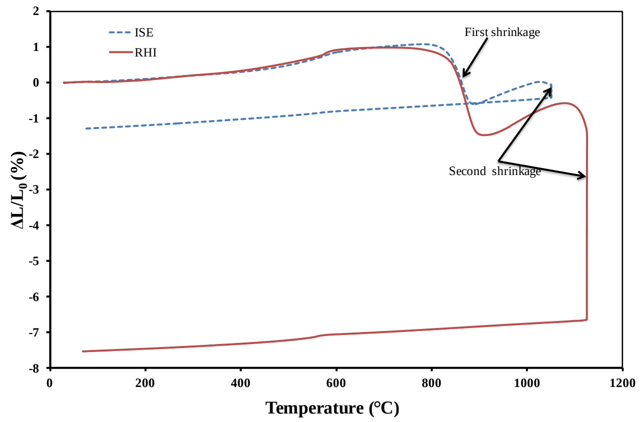


Fig 4: Dilatometric curve of ISE and RHI sediments.

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Volume 4, Issue 8, February 2015

The dilatometric analysis has pointed out the similarities and differences between the two sediments. ISE specimen exhibits less shrinkage than RHI specimen after firing at 1050°C for example. Taking into account our preliminary results [37], the mineralogical compositions and the shaping process, it is likely that ISE sediment may be consolidated at a lower temperature than RHI ones. Therefore, we have decided to fire RHI-based bricks at 1125°C and ISE-based bricks at 880°C, 1000°C and 1050°C. Since the plasticity of RHI green body appears problematic for extrusion, additional RHI-based compositions have been prepared by mixing RHI sediment with 10 and 20 mass% of RR40® clay (a commercial plastic clay supplied by Imerys Group). According to the supplier technical fact sheet, RR40® clay contains approximately 90% of kaolinite, 8% of quartz, and traces of anatase and rutile. The resulting compositions are labeled RHI-10R and RHI-20R with respect to 10 and 20 mass % of added RR40® clay. According to their dilatometric behaviors (supplemental file) and our previous work [37], the firing temperature of modified RHI bricks has been fixed at 1095°C.

The following firing conditions have been applied for bricks samples:

- a heating rate of 5°C/min from room temperature to the desired maximal temperature followed by a 2h dwelling at this maximal temperature;
- a cooling from the maximal temperature to room temperature at 10°C/min.

C. *Characterization of fired bricks*

The final densities and total porosity of the fired specimens are presented in **table 4** and **table 5**. The comparison of ISE and RHI fired samples shows that the porosity of RHI bricks fired at 1125°C is more important than that of ISE fired samples. In all cases, the porosity of sediment-based bricks (between 40% and 52%) is higher compared to the values obtained for clay-based roof-tiles (20%) and bricks (40%) [38]. Moreover, for theses sediments, the resulting fired bricks exhibit lower densities (between 1.37 et 1.52 g.cm-3) than those of most commercial fired-clay bricks (between 1.8 à 2 g.cm-3) [39]. The properties of use of fired products such as water absorption, flexural strength and thermal conductivity are expected to be related with these physical characteristics.

Table 4: Density and porosity of sediment-based bricks

Sample	Sintering temperature (°C)	Apparent density (g.cm^{-3})	Density (g.cm^{-3})	Porosity (%)
ISE	880	1.52	2.81	45.9
ISE	1000	1.37	2.82	51.4
ISE	1050	1.38	2.82	41.2
RHI	1125	1.37	2.75	50.2

Table 5: Properties of sediment-based bricks with 10 and 20% RR40® clay.

Sample	Apparent density (g.cm^{-3})	Density (g.cm^{-3})	Porosity (%)	Water absorption (%)	Thermal conductivity (±0.01 W.m^{-1}.K^{-1})	Ben stre (±0.5
RHI	1.37	2.75	50.2	28	0.66	7.5
RHI-10%RR40®	1.36	2.75	50.7	25.3	0.5	7.5
RHI-20%RR40®	1.48	2.72	45.4	19.8	0.6	8.7

Water absorption has been determined by using the mass variation of fired samples before (M1) and after 24h of immersion into deionized water at room temperature (M2). Finally, the calculation of water absorption was made by using equation (1).

$$Water absorption(\%) = \frac{M_2 - M_1}{M_1} \times 100 \qquad (1)$$

Fig 5 shows the water absorption values of ISE and RHI fired bricks. The obtained values range from 25% to 31% and are close to the requirements allowed for fired bricks applications [40]. Such high water absorption values can be justified by the level of total porosity in the studied samples. Nevertheless, the corresponding effective thermal conductivities (0.44 W.m^{-1}K^{-1} for ISE sample and 0.66 W.m^{-1}K^{-1} for RHI sample) are very satisfactory regarding a final application as construction fired bricks (**Fig 6**). The observed trend for both sediments is influenced by their porosity, but also by their mineralogical composition according to literature [41, 42].

Considering the bending strength of fired samples (**Fig 7**), it appears that the mean value is close to 8 MPa regardless of the sediment. Again, this value of bending strength is more appropriate for clay-based fired bricks (8 MPa) than for roof-tiles (13 MPa) applications.

The final characteristics of ISE and RHI samples fired at 1050°C and 1125°C respectively can be justified by the difference in microstructure as shown on **Fig 8**. RHI sample exhibits large pores with a loose structure that is detrimental for the properties of use. To this point, the final shrinkage should also be considered since it controls the dimensional stability, i.e. creep behavior of samples during firing. In the case of earthenware for building purpose, the shrinkage should be less than 2% and 1% for fired bricks and roof-tiles applications respectively [43]. Therefore, RHI sediment cannot be used alone as standard ceramic building products.

The addition of plastic clay to RHI sediments allow to reduce the shrinkage together with improving the plasticity of the green ceramic body (in relation with extrusion process). **Table 4** presents the final physical properties and properties of use for the modified RHI series. As expected, despite the high total porosity of fired samples, the water absorption and effective thermal conductivity are improved. Furthermore, the addition of 20 mass % of RR40® clay allows improving the flexural strength of RHI-based fired bricks from 7.5 MPa to 8.7 MPa.

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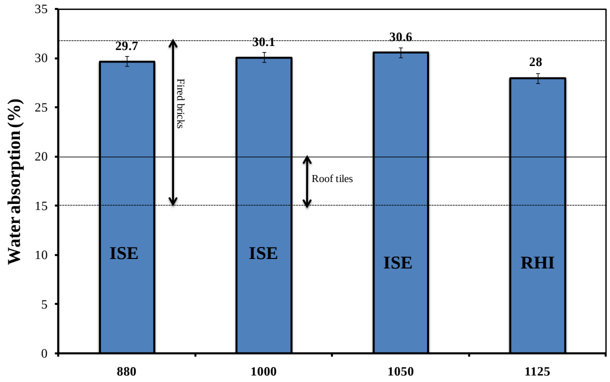

Fig 5: Water absorption of sediment-based bricks.

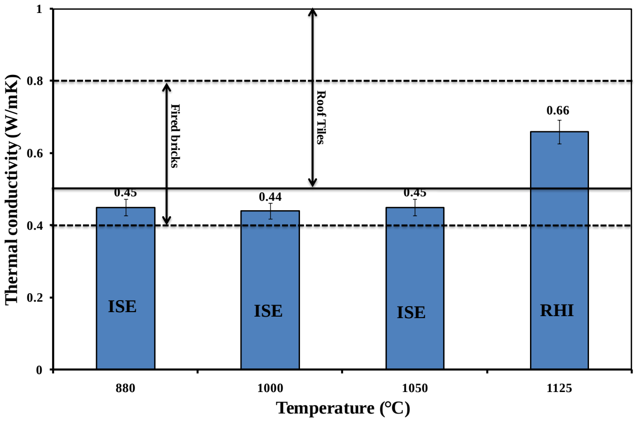

Fig 6: Thermal conductivity of sediment-based bricks.

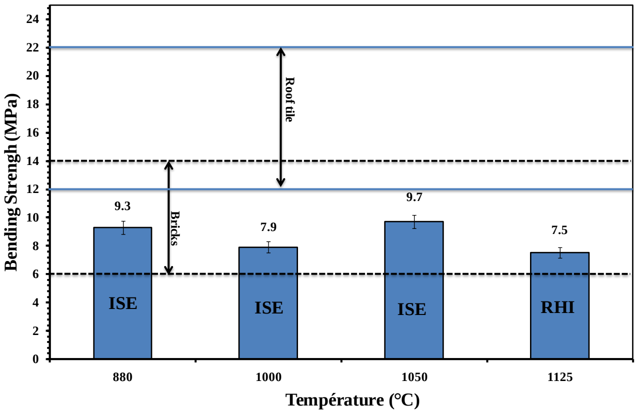

Fig 7: Bending strength of sediment-based bricks.

The observed behavior is assumed to result from the more densified microstructure of the fired bricks (**Fig 9**) and to an earlier consolidation thanks to the thermal transformations induced by the added clay. Actually, the SEM images (**Fig 9**) allow seeing the differences in pore distributions and the necking/bridging links between residual grains. In addition, XRD analyses of fired products (**Fig 10**) indicate the formation of high temperature phases such as wollastonite ($CaSiO_3$), gehlenite ($Ca_2Al[AlSiO_7]$), hematite (Fe_2O_3) and anorthite ($Ca[Al_2Si_2O_8]$) that may contribute to the strengthening of the fired samples.

To summarize, the present study has shown evidence towards the potential utilization of both sediments as raw materials for fired construction brick, and also revealed key parameters to tune in order to optimize the final characteristics of related fired products regarding silicate ceramics requirements for building purposes. According to literature, the chemical and mineralogical compositions of the studied sediments are suitable for bricks and tiles preparation [32]. However, it was necessary to conduct experiments in order to clearly identify the process ability drawbacks, such as the lack of plasticity for RHI sediment and the firing ability for both sediments. Larger amount of alkaline and alkaline earth elements induce lower firing temperatures. Besides, carbonates appear as ambiguous phases since their presence may on one hand help in lowering the firing temperature (low energy demand) and on the other hand promote a porous microstructure which is detrimental for the mechanical properties. A compromise should be found for the carbonates content also regarding the embodied-CO_2 of final products as required by environmental facts.

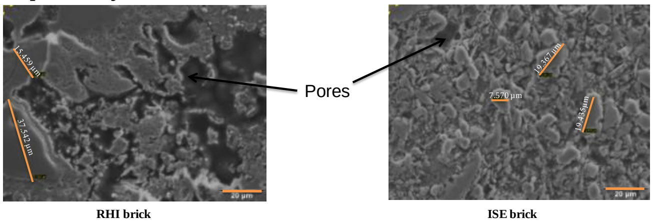

Fig 8: SEM micrographs of sediment-based bricks.

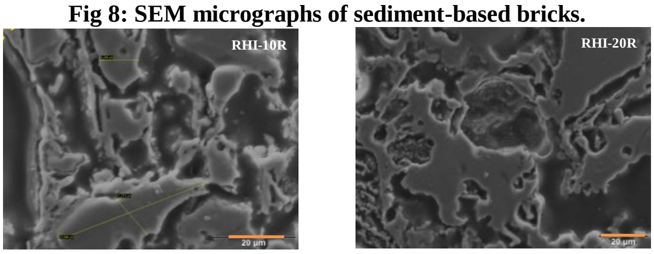

Fig 9: SEM micrographs of sediment-based bricks (RHI-10R and RHI-20R).

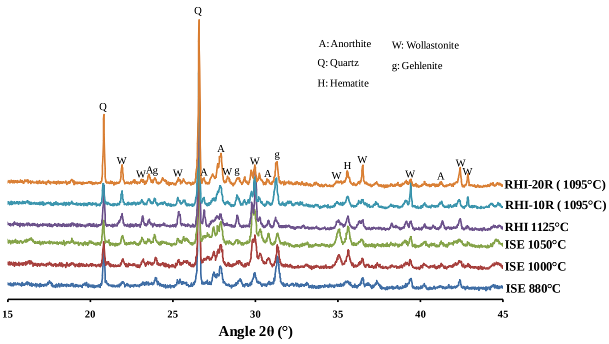

Fig 10: X-ray diffraction diagrams of sediment-based bricks.

IV. CONCLUSION

This work aims at showing the possible use of two sediments (ISE and RHI) from 2 particular areas in France, from the hydroelectric dam reservoirs of EDF Group in France for the production of ceramic materials.

Physical and chemical characteristics of these sediments are similar to those obtained for materials used in the traditional and standard ceramics industries. These sediment-based bricks exhibit a higher total porosity than conventional clay-based fired bricks. However, properties such as apparent density, water absorption, thermal conductivity and bending strength are close to those of ceramic bricks regarding current standards.

ISE sediment can be used without significant composition modification as traditional ceramic product and firing at 880°C.

RHI sediment exhibit many drawbacks regarding the plasticity (shaping using extrusion), the optimal firing temperature (1125°C), and the firing shrinkage (close to 7%) of related samples that may limit its utilization without significant modifications.

The addition of 10 mass% and 20 mass% of clay was made to RHI sediment to improve its plasticity and has also as a positive effect on the fired material properties. The bending strength increases of 7.5 MPa to 8.7 MPa and water absorption decreases from 28% to about 20%.

The X-ray diffraction showed the formation of phases such as wollastonite, gehlenite and anorthite within sediment-based bricks. Scanning electron microscopy of sintered samples showed a microstructure composed by isolated rounded pores, crystals with irregular shape and size, and a ceramic matrix surrounding the formed phases.

The results of this study show that the production of bricks from dam reservoir sediments is feasible and promising, since they can be used directly without additional grinding and at a relatively low firing temperature. It can be assumed that these sedimentary wastes could be a valuable renewable resource of raw materials in the manufacture of bricks. Further studies may involve a direct mixing of such sediment with typical commercial raw mixture commonly used for the industrial processing of fired bricks and tiles, in order to clearly assess the effective impact of sediment onto the properties of use of related fired products.

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