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Characterisation tests for mechanical stabilisation of dredged sediments using shell powder

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ABSTRACT

In port management and construction of port infra-structures, the deepening the seabed makes it necessary to dispose large quantities of dredged sediments. This problem is considerable not only in Italy: about 150 million m^3 of sediments are dredged every year in Europe. Both the national and international legislative framework, therefore, encourage the reuse, recycling, and recovery of dredged materials, even when contaminated, after adequate check and decontamination treatments. This scenario favours the scientific and technological research in the transformation of sediments from waste to environmental resource and several recent contributions aim to define scientifically based approaches for a systematization of sustainable reuse processes. This study is part of this context and entails experimental research that aims to contribute to open novel perspectives in this field, with applications for industrial interest. The results show the effects of the use of traditional binders for the treatment of fine-grained dredged sediments in comparison with greener solutions where part of the binder's content has been replaced by mussel shell powder. The note shows the results of the effects of these treatments on both the index and microstructural properties of the sediments.

Keywords: environmental geotechnics, dredged sediments, index properties, microscale, laboratory tests, geotechnical properties, shells, hydraulic binders, soil treatment.

1 INTRODUCTION

New strategies aiming to transform waste into novel by-products for other industrial applications are necessary to promote the sustainable use of resources. This principle, inspired by the fundamentals of the circular economy, is crucial in a world where the demand for resources is increasing exponentially to support continued economic and social growth and can be applied to various fields of Civil and Environmental Engineering, including the management and reuse of marine sediments. Marine sediments are dredged periodically (in Europe about 150 million m^3 /year) in harbour areas either for the port effectiveness or the site remediation (SedNet, 2011).

In this context, several research studies in geotechnical engineering entail the mechanical treatment of sediments by commercial cements and lime (Federico et al., 2015; Chang et al., 2007). Cements are the most widely used materials in the building industry (according to the WWF, concrete production could reach 5 billion tonnes by 2030) but they are also among the most harmful since concrete production process requires large amounts of energy and releases pollutants. For this reason, in the most recent years, novel strategies have been proposed making use of more sustainable binders that reduce the CO_2 emission and comply with the so called green new deal (Latifi et al., 2018; Roque et al., 2022; Paleologos et al., 2022). This note is part of this research, and it reports some results about an experimental study comparing standard solutions for the treatment of sediments and novel ones making use of lower content of binders in favour of the use of green additives. These original additives have been obtained by recycling mussel shells.

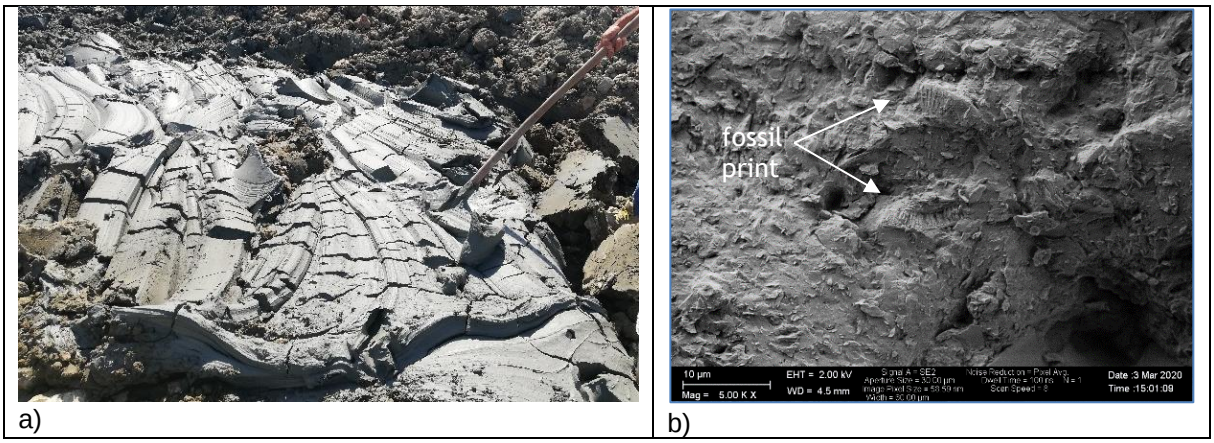
The shells are a biowaste currently produced in large quantities in Europe (i.e., about 550.000 tons in the past few years; FAO, 2023) and dumped in landfills with complex and expensive disposal procedures. The need to find out new strategies to minimise such waste is also demonstrated by the number of studies aiming to promote their reuse for more applications, i.e., from the cosmetic to the fertiliser industry and as natural additives to traditional binders (Papadimitrious et al., 2017; Morris et al., 2019; Paz-Ferreiro et al., 2012; Uster et al., 2014). However, to the best of the authors' knowledge, there are no studies presented so far making use of not calcinated shell powder in partial replacement of cements for the geomechanical treatment of sediments.

The main aim of this study is to present a solution for the mechanical improvement of dredged sediments by reusing shells, i.e., another waste of marine origin. Such solution could be valuable not only for the in-situ stabilisation of the sediments after dredging but also to realise new products (e.g., outdoor paving eco-bricks, breakwaters, embankments) from the new material obtained by mixing sediments, shell powder and cements. In the following, some laboratory results of microstructural and geotechnical tests carried out on dredged sediments treated with shell powder in partial replacement of different types of cements are presented. Moreover, experimental results of the same sediments when treated with just cements are also shown for comparison.

2 MATERIALS AND METHODS

2.1 Dredged sediments

Ports, lakes, and semi-enclosed water bodies require continuous dredging of sediments to guarantee proper navigation levels and to preserve the aquatic ecosystem (DelValls et al., 2004). Therefore, millions of tons of dredged sediments are generated worldwide every year (Mymrin et al., 2017) and their fate is a critical environmental concern. Among the strategies for their sustainable management, highly encouraged is their reuse as materials for engineering purposes. However, most of them exhibit extremely poor mechanical properties which make them unsuitable as construction materials in their current state.
As it commonly happens when dealing with natural soils, intergranular bonding and physic-chemical condition still affect the material behaviour (Mitchell, 1993; Lee, 2004). However, dredged sediments may lose their original geotechnical properties while they are being dredged, transported, and redeposited. Moreover, if the water content is high or increases during remoulding, they behave like a slurry. Also, high organic content, shells and diatoms, gas bubbles, fibrous materials and pollutants can affect their behaviour and possible reuse (Klein & Sarsby, 2000; Edil & Wang, 2000; Adamo et al., 2018). In general, the properties of fine-grained sediments can result highly variable, and their organic content typically generally spans from 0.4% to 11% (e.g., Shahri & Chan 2015; Ganesalingam et al., 2011; Lee et al., 2016). Most of them can be classified as CH (i.e., inorganic clay with high plasticity) in the Casagrande's plasticity chart. In view of the high complexity of these materials, the investigation of their composition as well as the characterisation of their physicochemical and geotechnical properties is still therefore an essential preliminary step towards the identification of the most proper hydro-mechanical or chemo-mechanical treatments (Vitone, 2019; Rakshith & Singh, 2016; Couvidat et al., 2018; Vitone et al., 2020, Paleologos et al., 2022; Cotecchia et al., 2022; Tang et al., 2021).



a) b)

Figure 1*. Untreated sediments, US: a) photo of the sampling site; b) SEM image.*

The untreated sediment studied (US, Figure 1a) has been dredged from the port of Taranto, in the south of Italy. Its microstructure was analysed through scanning electron microscope (SEM) after the freeze-drying of the samples. The SEM picture in Figure 1b, showing the presence of fossil prints in a random and chaotic fabric, is quite emblematic of the heterogeneous nature of marine sediments. The geotechnical characterisation of the dredged sediment presented in the following entails physical and plasticity properties that were determined according to ASTM standards. The carbon content was determined by dry combustion (DIN EN 15936) with an Analytic Jena MultiEA4000. Specifically, TOC was derived as total carbon content minus TIC. More details on the complexity of this natural material are reported by Sollecito et al. (2022).

2.2 Cements and shells

Three types of cements were used for the present study: from the traditional type I Portland Cement 52.5R (P), to more sustainable commercial binders, such as the type III Termocem green blast furnace cement (T) and the most recent Sulphoaluminate cement (CS). The latter two hydraulic binders result from a core process with < 550 kg /t CO_2 emissions and at least 30% of pre- or post-consumption recycled material. In this article, the results obtained using just type I Portland cement (P) and type III Termocem green blast furnace cement (T) are reported.

The mussel shell powder, MS, is a natural source of biogenic calcium carbonate, i.e., a *green* additive which here is reused to partially replace cement for mechanical treatment of dredged sediments. The shells used in this study are of the *Mytilus galloprovincialis* type (Figure 2a). For the production of the shell powder (Figure 2b), the mussel shells underwent the following treatment: i) washing with hot water for about 10 minutes and oven drying at 105 °C for 48 hours to remove impurities and some organic matter content (Othman et al. 2013); ii) milling in a Retsch jaw crusher; iii) sieving at 0.063 mm.

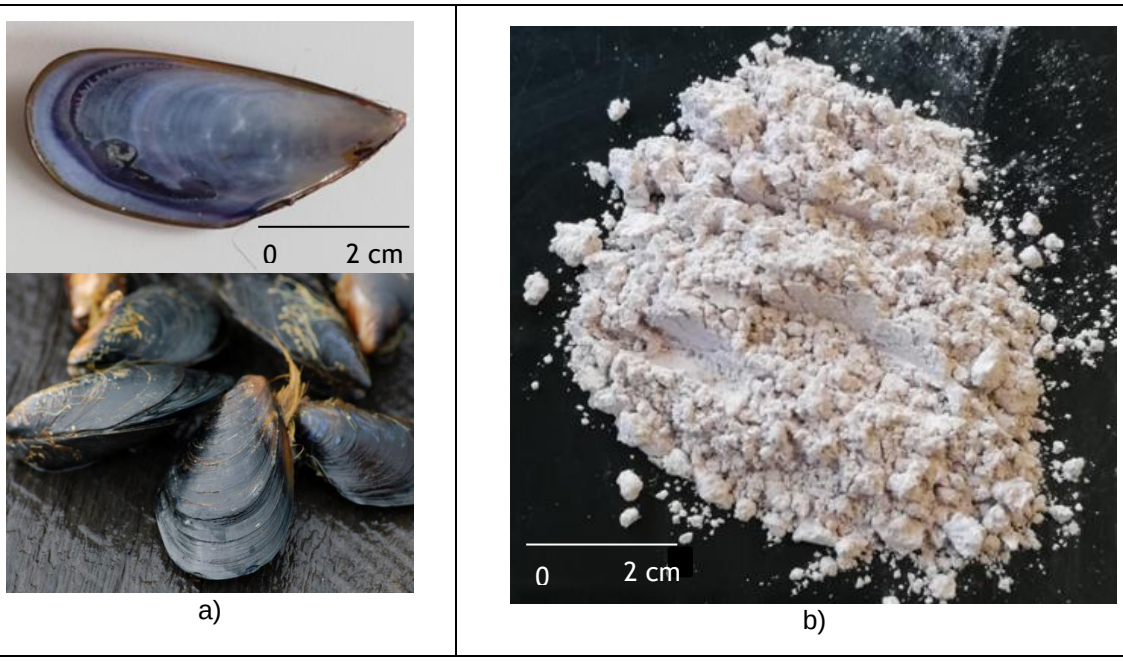


Figure 2. *Mussel shells, MS: a) the Mytilus galloprovincialis type of shells used; b) the powder produced.*

2.3 Preparation of the mixtures

The experimental programme entailed the comparison between mixtures formed by sediments and T or P cements and the corresponding mixtures where ¼ of P (or T) cement was replaced by mussel shell powder, i.e., P, T-shell powder. The content of binders added to the sediment was adjusted according to the dry weight of the sediment, which was determined from the natural water content of the sediment (w_0= 74.08%). The virgin material was first homogenised in a mechanical mixer for 10 minutes at medium speed (285 RPM). Subsequently, the binder was added, and the material was further mixed for 5 minutes at lower speed (140 RPM). After 7, 14 and 28 days of curing in marine water, the experimental programme involved not only the geotechnical characterisation of the mixtures, but also the investigation of their mineralogy, chemical and geochemical properties and their microfabric. To check the efficacy of

the original stabilisation solutions, an intensive experimental programme has been conducted on the dredged sediments mixed with both traditional and cement-shell binders, for comparison. The chemical composition of cement and mussel shell powder was performed with XRF analyses. In addition, the fabric of the treated sediments has been studied by means of SEM analysis on both untreated and treated sediments samples. For all the tests the Zeiss leo-1530 apparatus was used, and the freeze-drying method and Tungsten coating were performed for all the sediment samples.

3 RESULTS AND DISCUSSION

3.1 Characterisation of base-materials

3.1.1 Dredged sediments

The untreated sediment (US) is a clayey silt of negative consistency (CI= (w_L-w_0)/PI= -0.71) that can be classified as high plasticity soil, CH (USCS, ASTM 2011) (Table 1 and Figure 3). As suggested by both standards and literature (e.g., BS 1337; Sollecito et al., 2019; Sollecito et al. 2022), the water content w and the Atterberg limits were corrected to take account of the pore fluid salinity. Moreover, Atterberg limits have been determined on the total sample, following an unconventional procedure (UP; full square in Figure 3), instead of on the material passing on the ASTM sieve n. 40, according to the ASTM procedure (ASTM; empty square in Figure 3). This allowed considering the effect of the presence of algae and other organic materials that actively interact with the soil particles (e.g., Roque et al., 2022). In detail, such organic material is essentially represented by algae, and it is retained to the ASTM sieve n. 40 (photo ASTM procedure in Figure 3).

In detail, Figure 3 shows that, when the limits are determined on the total sample, both liquid limit ad plasticity index increase (i.e., w_{L_UP}= 53%; PI_{UP}= 28%; w_{L_ASTM} = 46%; PI_{ASTM}= 23%), so that the US passes from being classified as CL to CH soil (USCS, ASTM D2487-17e1). The distance between the two points in the Casagrande's plasticity chart is a first quantification of the active role of these organic fractions in interacting with the clay particles to modify the sediment plasticity. The consistency index of the sediment is always negative and equal to -0.71 and -1.2 if computed by using either the Atterberg determinations on the total materials or following the ASTM procedure, respectively. Table 1 also reports the specific gravity of the soil solid (G_s) and the amount of organic matter measured in US by total organic carbon (TOC).

The chemical characterisation of the sediment (ISPRA, 2010) showed that heavy metals and organic pollutants are present in lower quantities than regulations limits (Legislative National Decree 152/06).

Table 1. *Physical properties of untreated sediments, US. Key. γ_s: weight of soil solids per unit volume; SF: sand fraction; MF: silt fraction; CF: clay fraction; TOC: total organic carbon; Gs: specific gravity of soil solids, w_0: natural water content.*

Material	**γ_s [kN/m^3]**	**SF [%]**	**MF [%]**	**CF [%]**	**TOC [%]**	**Gs [-]**	**w_0 [%]**
US, Untreated sediment	15.95	5.80	63.80	30.40	1.52	2.68	74
Standard used	ASTM D 854-14	ASTM D422			DIN EN15936	-	ASTM D2216-D4542

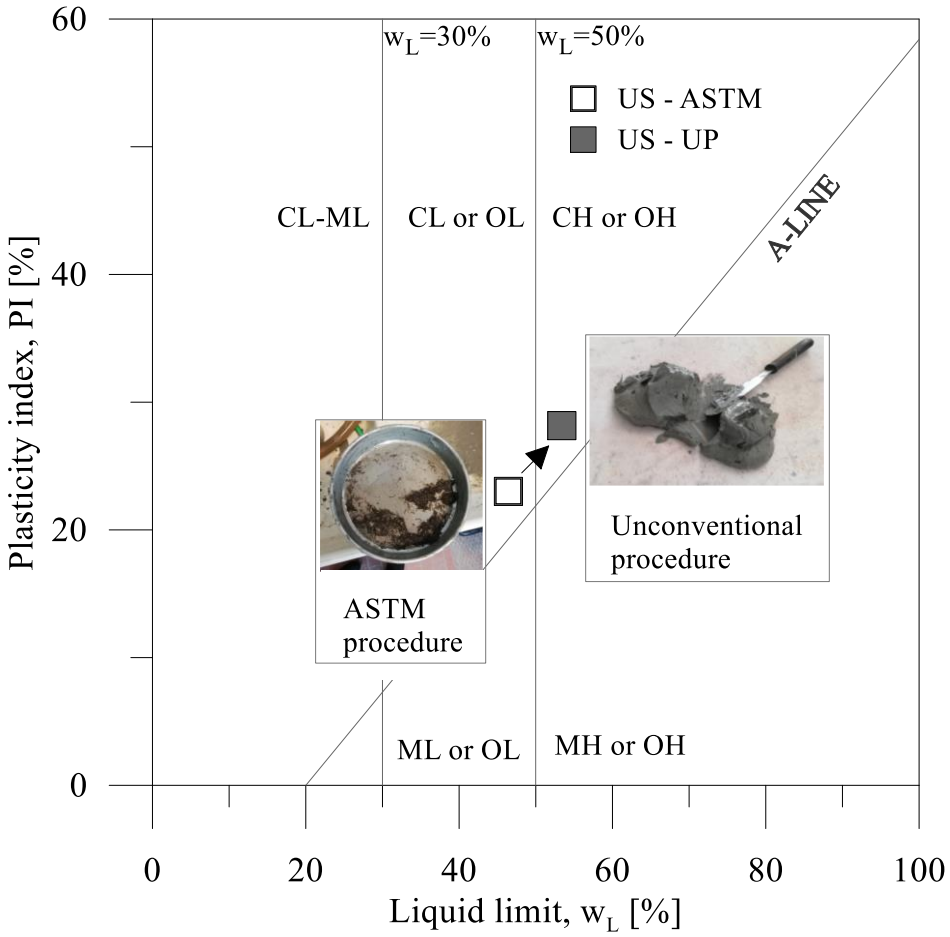


Figure 3. *Casagrande plasticity chart of marine sediment from the Port of Taranto.*

3.1.2 Cements and mussel shell powder

The chemical composition of P and T cement is reported in Table 2. The data show that the principal components differences in the chemical composition of the cements are the higher percentages of CaO, Fe_2O_3 in P and higher percentages of Al_2O_3, SiO_2 and MgO in T.

The particle size distribution of the mussel shell powder used in this study is characterised by D_{50} = 6.32 μm and C_u = 2.213. Table 2 shows that chemical composition of the mussel shell powder that, as expected, is mainly composed by Calcium (53.61 wt% expressed as CaO) with small fractions of other oxides. In Figure 4 a SEM image of mussel shell fabric is shown. Differently from geogenic limestone, where $CaCO_3$ aggregates consist mainly of rounded particles, the fabric of mussel shells is made up of elements of similar average size (2–6 μm) but more elongated shape. Moreover, as reported in the literature, the structure of mussel shells, like all bivalves, can be divided into three parts: the outer layer, periostracum, the middle layer, i.e., the prismatic layer (Gènio, 2014), and the inner layer referred to as nacre (Martínez-García et al., 2017). Elongated shapes characterise both prismatic layer and nacre. The first is the thicker one (around 400 μm; Hahn, 2011) and its structure is composed by arrays of parallel and very elongated calcite prisms (Gènio et al., 2014), which are polygonal in cross section. Also, the nacre, i.e., a biomineralized composite (Hahn, 2011) about 10 μm thick, comprises laminar aragonite which is also oriented parallel to the surface and held together by an organic matrix composite of polysaccharides (chitin), proteins and glycoproteins. Despite the heating process, the SEM images of the mussel shell powder not only shows the presence of rhombic calcite but also the persistence of the lamellar aragonite (Figure 4).

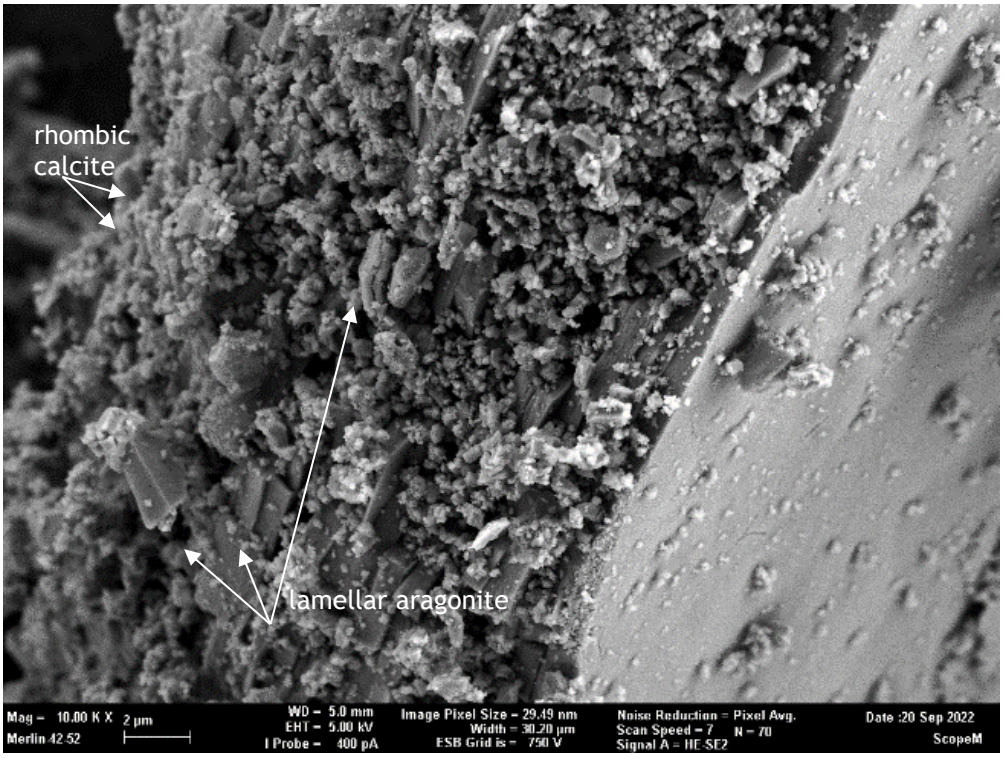


Figure 4. *SEM photograph of the mussel shell powder.*

Table 2. *Chemical composition of Portland cement, P, Termocem green cement, T, and mussel shell powder, MS.*

Oxides [% mass]	**P**	**T**	**MS**
CaO	63.06	44.9	53.60
Al_2O_3	3.92	8.25	0.02
Fe_2O_3	4.51	1.48	0
SiO_2	18.09	32.81	0.038
MgO	1.59	5.79	0.239
Na_2O	0.5	0.52	0.355
K_2O	0.93	0.81	0.034
SO_3	4.13	3.61	0.197
P_2O_5	0.11	0.05	0.034
SrO	0.03	0.09	-
MnO	0.07	0.15	-
TiO_2	0.22	0.38	0
Mn_2O_3	-	-	0.002
Sr [PPM]	-	-	812
Ni [PPM]	-	-	48
Cu [PPM]	-	-	118
LOI	2.64	1.27	45.58

3.2 SEM photographs of treated sediments

Figure 5 shows the microphotographs at magnification 5.00Kx of the different mixtures. In detail, the SEM photographs of the sediments treated with 8% of P and T cements (US8P in Figure 5a and US8T in Figure 5c) and the same sediments where 2% of P or T cement in the mixtures is replaced by 2% of mussel shell powder (US6P2MS in Figure 5b and US6T2MS in Figure 5d) are reported.

The presence of calcium hydroxide plate, CH plate, and calcium silicates hydrates formation, CSH, is evident not only from the micrographs of the sediments treated with 8% of P cement (US8P, Figure 5a) but also on that treated with 6% of P cement and 2% of mussel shell powder (US6P2MS, Figure 5b).

Moreover, the needle forms of CSH are arranged into “honeycomb” structures and, in some cases, they fill, with other cement products, the spaces produced by Foraminifera shells.
Also, the treatment of the sediment with 8% of T cement (US8T) produces calcium hydroxide, CH, whose presence is confirmed with its platy morphology in Figure 5c. Finally, Figure 5d shows that in the fabric of the US6T2MS sample, the mussel shell powder appears completely encapsulated in the cement-sediment structure, acting as a connector thanks to the typical elongated shape of the mussel shells.

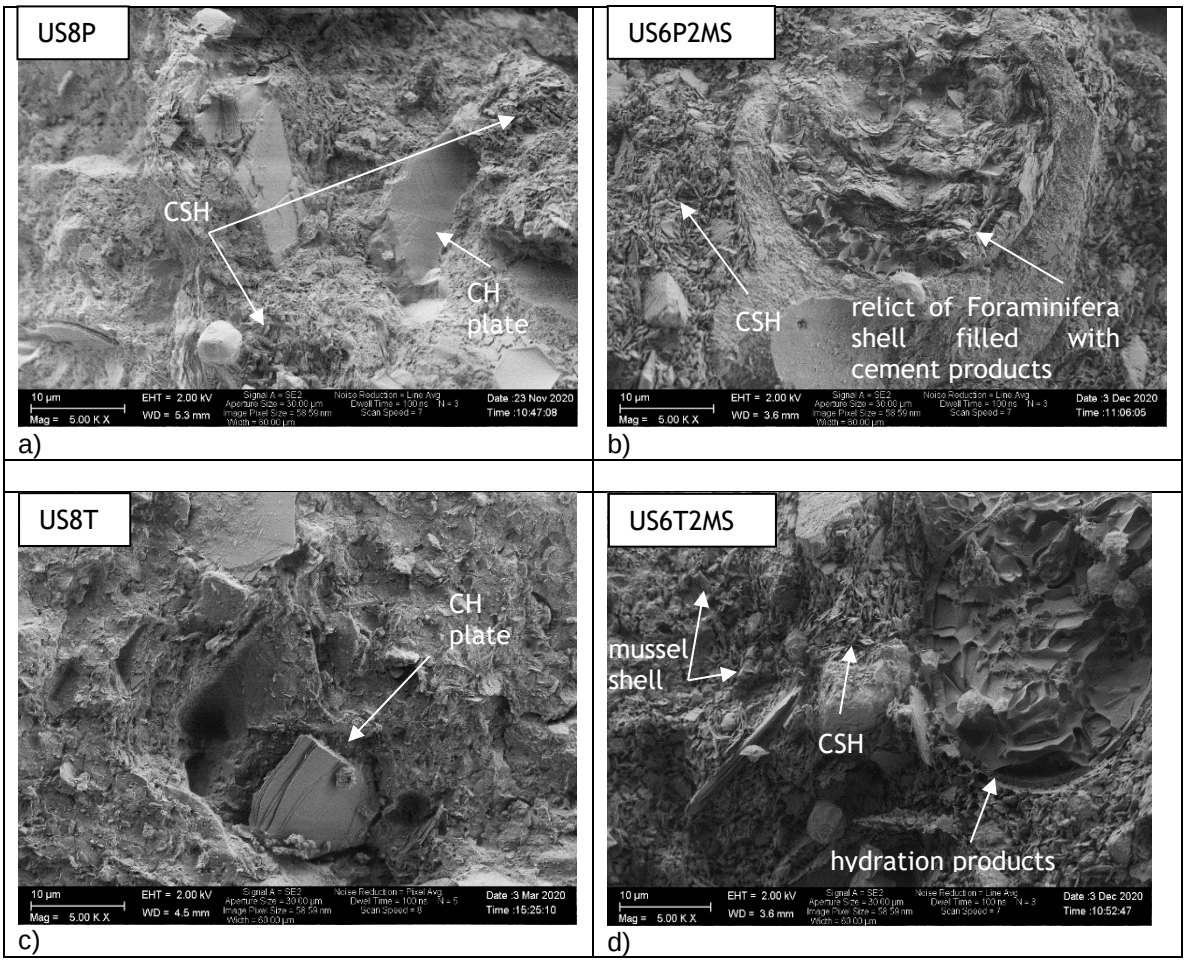


***Figure 5**. SEM photograph of a) sediment treated with 8% of P cement, US8P; b) sediment treated with 6% of P cement and 2% of mussel shell powder, US6P2MS; c) sediment treated with 8% of T cement, US8T; d) sediment treated with 6% of T cement and 2% of mussel shell powder, US6T2MS.*

3.3 Atterberg Limits and Consistency of treated sediments

Table 3 reports the Atterberg limit, water content and consistency index values that were determined on the natural sediments and on the sediments treated with P, T and the corresponding cement-shell mixtures after 28 days of curing. Moreover, in Figure 6 the Casagrande plasticity chart is reported including such Atterberg limit determinations.

The results in the figure show that, if 8% of any additive is used, 28 days of curing are sufficient to transform the untreated sediments (US) from CH (clay of high plasticity) to MH soil (inorganic silt of high compressibility and organic clay), according to USCS classification (USCS, ASTM D2487-17e1). In particular, after 28 days of curing the plasticity index of US8T is slightly lower than that of US. Instead, the treatment with 8% of P cement, US8P, produces a small increase in PI with respect to the corresponding value of untreated sediment.

The use of mussel shell powder (i.e., mixtures US6P2MS and US6T2MS) produces an increase in the PI value, that is higher the values recorded in US8P (or US8T) and US samples. Moreover, the limit

liquid increases in all the mixtures (with and without MS) with respect to the corresponding value determined on the untreated sediment sample.

Finally, the reduction in water content brings about an increase in consistency of more than 100% for all the mixtures, which move from fluid to plastic consistency (Table 3). Replacing part of the P (or T) cement with MS (i.e., US6P2MS, US6T2MS) not only does not seem to affect the results but causes a further (even if small) increase in consistency (about 5%).

Table 3. *w_L, w_0 and PI values of US, P, T-cement and P, T-shell powder treated sediment samples after 28 days of curing.*

Materials	w_L [%]	PI [%]	W_0 [%]	CI [-]
US	53.59	28.61	74.00	-0.71
US8T	83.26	26.51	67.82	0.58
US8P	86.50	32.20	66.95	0.61
US6T2MS	86.42	32.66	66.48	0.63
US6P2MS	85.17	29.83	66.47	0.63

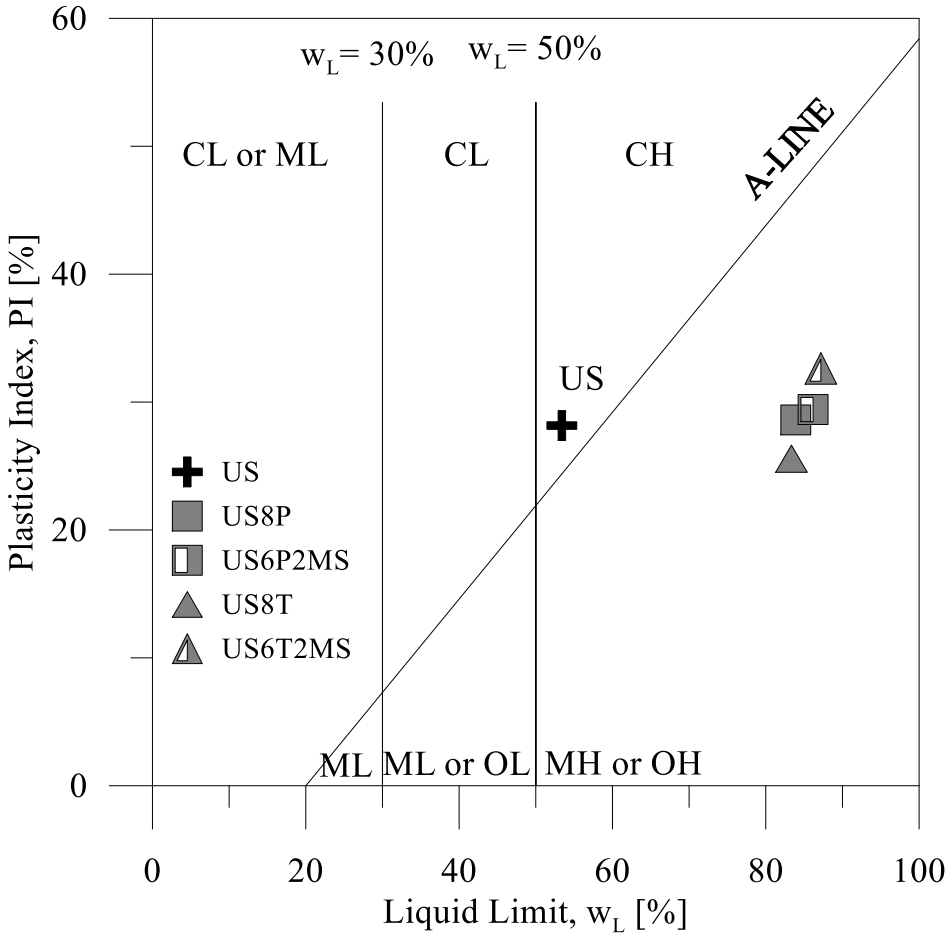


Figure 6. *Casagrande's plasticity chart of US P, T-cement (US8P and US8T) and P, T-shell powder (US6P2MS and US6T2MS) treated sediment samples after 28 days of curing.*

4 CONCLUSIONS

The note shows some multiscale experimental results about the use of mussel shell powder as a green additive to partially replace different kinds of cement in the mechanical treatment of dredged sediments. The sediments used were sampled in the south of Italy and well represent, for their composition, consistency and heterogeneity, fine grained soils typically dredged from port areas, in particular

concerning fluid consistency, high plasticity, and organic content. The treatment of the sediments was carried out both with commercial binders (type I Portland cement, P, and type III Termocem green blast furnace cement, T) and with a new mixture produced by partially replacing the same cements with the corresponding content of mussel shell powder (MS), appropriately treated without calcination.

The data show that, after 28 days of curing, if part of P (or T) cement is replaced by MS the performance of the mixture MS-sediment-cement is better than that of sediment-cement mixtures in terms of consistency index. Moreover, the SEM analysis provides evidence that, together with their chemical role as source of biogenic calcium, the mussel shells, also thanks to the typical elongated shape of aragonite, are mechanically supporting the connections within the cement-sediment fabric as completely encapsulated therein. Moreover, the presence of hydration products, i.e., calcium silicates hydrates formation, CSH, is evident from the SEM photographs. While limited to one particular type of marine sediment, this research demonstrates significant potential of using mussel shell powder as a partial replacement of cement on the improvement of the mechanical properties of fine-grained sediments. Future developments of this research are ongoing to further deepen the geotechnical properties of the sediment mixtures and the possible role of the mussel shell powder as adsorbent of pollutants in sediments contaminated by either organic and/or heavy metal pollutants.

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