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Valorization of a Highly Organic Sediment: From Conventional Binders to a Geopolymer Approach

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Abstract: The objective of this research is to investigate the possible reuse of dredged sediments from the port of Cherbourg, France, as an alternative material in road engineering and as a backfill material. These dredged sediments contain high percentages of organic matter (OM), and the presence of OM in the sediment, even in small amounts, can affect the engineering properties of sediments. This research was carried out in two series: the sediment was treated with traditional hydraulic binders (ordinary Portland cement (OPC), calcium sulfo-aluminate (CSA) cement, quarry sand (QS), lime, and a combination of them) in the first series, and with pozzolanic binders in the second series (ground-granulated blast-furnace slag (GGBS) and fly ash (FA)), along with the introduction of an activator. According to French legislation, these two pozzolanic binders (GGBS and FA) have no carbon footprint as they are industrial by-products, and therefore, the second series of this research is considered to be highly eco-friendly and economical. Sediment treated with hydraulic binders yielded a maximum value of unconfined compressive strength (UCS) of 1 MPa at 28 days. Out of eight formulations made using traditional binders, only one formulation barely met the French criteria to be used in the sub-base layer of roads. The development of geopolymer using alkali-activated GGBS and then the incorporation of 30% sediments yielded a UCS value above 2 MPa at 28, 60, 90, and 180 days. Furthermore, the addition of 5% lime and 3% granular calcium carbonate in the same mixture (geopolymer + 30% sediments) increased the UCS by up to 60% and 90%, respectively.

Keywords: dredged sediment; valorization; geopolymer; alkaline activator; highly organic matter content; GGBS; fly ash; conventional binders

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1. Introduction

Dredging produces considerable amounts of sediments throughout the world. France, for example, generates about 50 million cubic meters of these sediments per year [1]. Most of the time, these sediments are considered as waste due to their poor engineering properties. Therefore, a handsome amount of money and time is needed to dispose of them without affecting nature too much. To reduce the climate impacts of the construction work, it is necessary to increase the resource efficiency by reusing waste material [2]. Valorization of dredged sediments by utilizing industrial by-products is regarded as a highly eco-friendly and economic approach. It means saving raw materials and, incidentally, natural resources, but it also means eliminating the waste. Material recovery has a cost. Reducing it by eliminating one or more wastes, such as dredged sediments and industrial by-products, has given rise to the concept of co-valorization, which is both economic and ecological: eliminating waste and saving natural resources.

The management of dredged sediment (polluted or not) has become an environmental and economical concern for today's world. Moreover, with the new European Union directives [3–5], harbor and dam managers are encouraged to find environmentally friendly

solutions for these materials. The potential recycling options for sediments are well-known (Figure 1), ranging from direct recycling (unpolluted sediments) to energy-intensive processes such as firing (bricks and tiles) or incineration (calcined sediments).

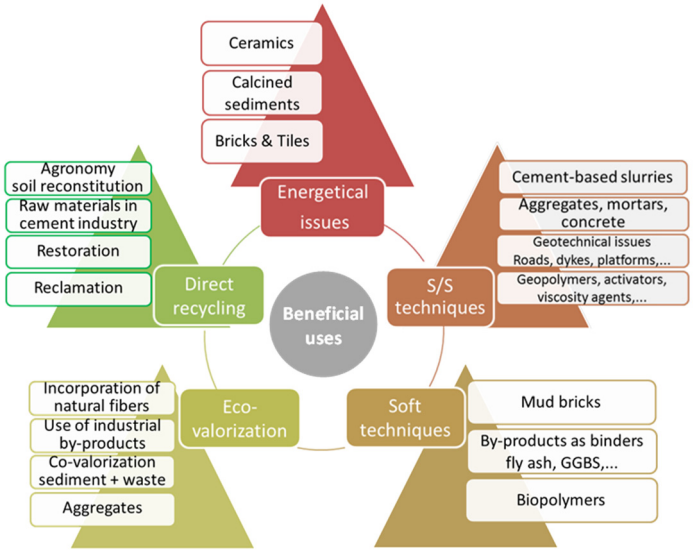


Figure 1. Potential recycling options for dredged sediments.

The recycling of sediments as a material has been the topic of numerous studies [6] but large-scale applications have remained very limited. There are different explanations for these limitations. The high-water content of the dredged sediments does not allow for their transport or direct reuse, and dewatering is necessary. Furthermore, if the sediments are contaminated, they are subject to strict environmental monitoring according to the laws in force in each country and the recovery methods are difficult to implement. However, the technique of stabilization/solidification using hydraulic or conventional binders has been widely used, and then industrial by-products of varying degrees of reactivity (fly ash, slag) have been added to these binders in an attempt to limit the dosage of cement, which emits CO_2. The low dosages of binders are sometimes provided with granular corrections (sand) to obtain sufficient strength for the reuse of sediments treated with these binders and additives in backfill or road sub-base materials. In practice, however, water must be removed from the sediments before any treatment. Moreover, the presence of organic matter significantly reduces the desired strength. Organic matter inhibits hydration reactions. The recovery of dredged sediment is very complex but the ongoing research on this topic is improving the concerned knowledge day by day. A number of studies throughout the world have expressed the scope of valorization of dredged sediments in the bricks and ceramic industry [6–10], in the cement industry [11–16], as well as in road engineering [17–24]. Hydraulic binders (cement and lime) with a high carbon footprint have been and still are the most widely used, even though there is currently a trend towards a reduction in their usage. The incorporation of pozzolanic binders (slag, fly ash) with or without the addition of cement and with or without the use of particle size correctors has been the purpose of many studies [23–27]. The direct use of pozzolanic binders such as slag and fly ash can stabilize sediments to relatively acceptable characteristics.

The construction sector is facing progressive depletion of natural resources. The escalating demand of traditional hydraulic binders is making the natural resources difficult to access. The manufacturing of the cement requires huge quantities of non-renewable resources, i.e., raw material and fossil fuels. It is estimated that cement production generates almost 6% of total carbon dioxide produced by human activity [28].

Recently, the idea of substituting the traditional cement with activated by-products in the recovery of sediments has emerged [29]. However, fewer studies have been carried

out on the valorization of sediments using activated by-products, and by analyzing the activation processes used, it is necessary to calcine the sediments beforehand [30–35] or, once the sediment-based mixtures have been activated, the curing process requires a passage in an oven at high temperature and for varying lengths of time [32,34,36]. Although the strength of the obtained materials could find various applications in construction, they are not eco-friendly materials because their production is energy-intensive, the dosage of activators remains high, and their cost is not evaluated [37]. However, the use of geopolymers in the valorization of sediments is promising as the activation can take place directly in the sediment-precursor mixture at room temperature, with a low temperature during curing time [32,38]. This study focuses on the recovery of highly organic marine sediments in filling material, backfill, or road layers using conventional binders as well as alkali-activated industrial by-products.

The incorporation of industrial by-products such as slags and ashes instead of OPC seems interesting, as it allows the co-valorization of two wastes (dredged sediments and industrial by-products) and thus to eliminate them both. Pozzolanic binders (with alkaline activator) can evince mechanical properties [39] and thermal stability [40,41], as compared to OPC. Sediment and additions of fine-grained industrial by-products with binding properties, with or without the use of activators, can provide good stabilization and solidification results [27,42]. One example of sediment co-valorization is the work of Silitonga [43], which introduced the addition of ash and silica fumes into mixtures of contaminated sediments to make a road sub-base material. Finally, co-valorization of sediments means either associating sediments with other waste-like materials to enable their valorization or reducing the treatment cost of sediments by utilizing other waste-like materials.

The main purpose of this research is to recycle a marine sediment with a high organic matter content as an alternative material, knowing that conventional hydraulic binders will have difficulty stabilizing these sediments due to the presence of these organic matters (>15%). The dredged sediments from the port of Cherbourg are valorized using both traditional hydraulic binders (OPC, lime, CSA cement) as well as pozzolanic binders (GGBS, FA), and the results are compared. The alkali activation of the pozzolanic binders was performed with an activator (NeoliX®). NeoliX is the activator, and GGBS is the precursor. The mix GGBS-NeoliX constitutes a geopolymer and the mix GGBS-NeoliX-sediments is the geo-composite. The activator is developed and provided by a French company (Neolithe, Chalonnes-sur-Loire, France; https://neolithe.fr/, accessed 13 May 2022). To limit the CO_2 emission, the purpose of the paper is to promote the use of geopolymers, and in practice it shows better mechanical results as compared to high-carbon traditional binders.

2. Materials and Methods

2.1. Dredged Sediments

Sediments were dredged from three different points, shown by the red circles in Figure 2, at the port of Cherbourg, Normandy, France.

Dredged sediments at points 1, 2, and 3 are termed as CHER-1, CHER-2, and CHER-3, respectively. The characterization of the sediments is the first step in any recovery process. It allows to determine a set of properties necessary to choose a valuation chain to optimize this process. Therefore, the physicochemical characteristics of sediment, the contaminants' interaction with the sediment matrix, the amount of sediment, as well as local environmental factors, must all be considered when designing an appropriate sediment recovery strategy [44]. Table 1 outlines the geotechnical tests performed on the dredged sediments, the standard, as well as the method used to determine each characteristic.



Figure 2. Sample collection: exact locations at the port of Cherbourg, Normandy, France.

Table 1. Testing methods and standards used to obtain characteristics of dredged sediments.

Type of Test	Methods and Tests	Test Standards	Findings
Sieve analysis	Laser diffraction method	ISO 13320, 2020	Cu, Cc, D_{max}
Density	Helium gas pycnometer method	DIN 51913, 2013	γ_s (g/cm^3)
Clay contents	Methylene blue adsorption test	NF P94-068, 1998	MBV (g/100 g)
Water content	By heating in oven at 60°	NF P94-050, 1990	W (%)
Atterberg Limits	Liquidity at the cup/plasticity with a roll	NF P94-051, 1993	LL, PL, PI, CI (%)
Organic matter	Method by calcination	NF XP P94-047, 1998	OM (%)
Carbonates	Calcimeter method	NF P94-048, 1996	$CaCO_3$ (%)
Compaction	Miniature Proctor test	NF P94-093, 2014	MDD, OMC

The characteristics of dredged sediments from the three locations at the port of Cherbourg were obtained following the French standards, and the results are presented in Table 2. Each recorded result is the average of three samples.

Experimental data by Hamouche and Zentar [45] show that when the OM increases from 5% to 15%, the highest OM sample loses around 60% of its bearing capacity compared to the raw specimen. Other studies have found an increase in optimal water content and a decrease in optimal dry density with an increase in OM in the dredged sediments [46,47], as well as an increase in the liquidity plasticity limit with an increase in OM [48,49]. The presence of large amounts of organic matter can cause problems regarding the cement hydration [50]. Sediments with high organic matter contents cause disruptive effects in hydraulic binders as well as pose problems for smooth embankment [51–53]. As per GTR recommendations [54], A1 and A2 materials having OM greater than 3% must be the subject of specific studies to evaluate their behavior. In fact, the organic matter is an undesirable component in a material of construction [17]. Therefore, determination of organic matter content is one of the most important steps in valorization studies. The sediment from Cherbourg contains a high amount of organic matter content (Table 2), and the presence

of organic matter decreases the pH as microbial degradation of organic matter produces organic acids. The addition of lime in sediments increases its pH and therefore permits the activation of the pozzolanic activity by producing $Ca(OH)_2$. Using the lime fixation method, the quantity of lime to reach high constant pH can be adjusted. Several studies have already been conducted on the sediment stabilization using lime [55–59].

Table 2. Physiochemical characteristics of dredged sediments.

Characteristic	Symbols	CHER-1	CHER-2	CHER-3	Used Standard
Maximum particle size	Avg. D_{max} (mm)	20	20	20	ISO 13320-1, 2020
Size under 80 µm	Avg. Size < 80 µm (%)	>70	>70	>70	ISO 13320-1, 2020
Size under 2 mm	Avg. Size < 2 mm (%)	>99	>99	>99	ISO 13320-1, 2020
Coeffient of curvature	Avg. C_c	10.29	11.78	12.91	ISO 13320-1, 2020
Uniformity coeffient	Avg. C_u	0.93	0.95	0.97	ISO 13320-1, 2020
Dry density	γ_s (g/cm^3)	2.27	2.34	2.25	DIN 51913, 2013
Methylene blue value	Avg. MBV (g/100 g)	1.62	1.59	1.61	NF P94-068, 1998
Natural water content	Avg. w_n (%)	>150	>150	>150	NF P94-050, 1990
Plasticity index	Avg. PI (%)	20.84	22.97	23.53	NF P94-051, 1993
Organic matter content	Avg. OM (%)	16.75	15.68	19.68	NF XP P94-047, 1998
Carbonates	Avg. Carbonates (%)	8.25	3.66	2.35	NF P94-048, 1996
Maximum dry density	MDD (g/cm^3)	1.43	1.38	1.41	NF P94-093, 2014
Optimum moisture content	OMC (%)	27.50	28.00	33.50	NF P94-093, 2014
Relation between w_n and OMC	Relation w_n and OMC	w_n > 1.3OMC	w_n > 1.3OMC	w_n > 1.3OMC	GTR, 2000
Soil class	Soil class	A2	A2	A2	GTR, 2000
Humidity nature	Humidity nature	Very humid	Very humid	Very humid	GTR, 2000

The percentage of sand, silt, and clay was obtained from grain size classification (NF X31-107, 2003). As per sediment texture studied after Soil Survey Staff (1951) [60], the particles with diameters of <2 µm, 2–63 µm, and 63 µm–2 mm are clay, silt, and sand, respectively. Sediments from Cherbourg contained very little clay, but high quantities of silt and sand. The sediments contained low concentrations of lead (Pb), zinc (Zn), and vanadium (V), as determined using the method of optical emission spectroscopy with high-frequency-induced plasma following French standard NF EN ISO 11885, 2009 (Table 3).

The presence of heavy metals in the dredged sediments can reduce the efficiency of these sediments. The environmental quality of sediments is assessed using the Decree of "9 August 2006" [61], which compares heavy metals to the N1 and N2 thresholds. Below level N1, potential impacts are negligible, between N1 and N2, further investigations are recommended, and above the N2 level, further investigations are necessary. As all the heavy metal concertation was below the N1 threshold limit for each of the three sediments, no further investigation to tackle these heavy metals is recommended by French regulations.

Table 3. Heavy metal concentration (part per million or mg/kg) and percentage of clay, silt, and sand contents in the dredged sediments.

Samples	Cd	Ni	Pb	Cu	Zn	Hg	As	V	Cr	Cu
CHER-1	0.16	13.53	70.89	22.40	223.74	0.32	7.79	54.43	62.7	22.40
CHER-2	0.18	14.01	69.10	20.11	221.80	0.41	8.50	60.12	60.8	24.60
CHER-3	0.20	13.43	70.15	23.96	225.12	0.25	12.10	41.00	58.9	20.50
N1	1.20	37.00	100.00	45.00	276.00	0.40	25.00	75.00	90.00	45.00
N2	2.40	74.00	200.00	90.00	552.00	0.80	50.00	120.00	180.00	90.00

Nature	Clay (%)	Fine Silt (%)	Coarse silt (%)	Fine sand (%)	Medium sand (%)	Total silt (%)	Total sand (%)
CHER-1	6.60	47.40	26.00	19.10	0.90	73.40	20.00
CHER-2	5.10	44.40	20.30	20.60	9.60	64.70	30.20
CHER-3	4.81	38.69	23.71	25.50	7.29	62.40	32.79

2.2. Low-Carbon (Pozzolanic) Binders

2.2.1. Ground-Granulated Blast-Furnace Slag (GGBS)

GGBS (commercial name Ecocem®) used in this study was manufactured and provided by a company named Ecocem France SAS, and is certified in accordance with the requirements of standard NF EN 197-1, 2012. GGBS, provided by Ecocem France, is composed mainly of oxides of calcium, magnesium, aluminum, silicate (Table 6), less than 1% crystalline silica, and less than 2 ppm water-soluble chromium VI. The physical characteristics of GGBS used in the study are tabulated in Table 4.

Table 4. Physical properties of GGBS.

Characteristic	Numerical Value
Solubility in water (T = 20 °C)	<1.5 g/L
Relative density (specific gravity)	2.85–2.95
Bulk density	0.95
pH (T = 20 °C in water)	9.8
Boiling point	>1250 °C
Stability (expansion)	1 mm (according to NF EN 196-3, 2017)
Set start time	220 min (according to NF EN 196-3, 2017)
Activity index at 28 days	98% (NF EN 196-1, 2016)
Specific surface area (SSA)	4005.80 cm^2/g

Source: Technical data sheet provided by Ecocem France SAS.

2.2.2. Fly Ash (FA)

FA (commercial name Sodeline®) is fluidized bed ash from the Emile Huchet power station located in the department of Moselle, France. FA for this study was provided by the French company named Surchiste. Table 5 presents the physical characteristics of the FA used in this research.

Table 5. Physical characteristics of FA.

Characteristic	Numerical Value
Origin	bituminous coal
Activity index at 28 days	70% to 80% (NF EN 196-1, 2016)
Density	1.48 g/cm^3
pH	10
Specific surface area (SSA)	1811.50 cm^2/g

Source: Technical data sheet provided by Surchiste France.

2.2.3. Activator (NeoliX®)

NeoliX® is an activator developed by Neolithe company under a patent for using it in the process of manufacturing of aggregates from waste. The advantage of the Neolithe activator is precisely that this activator is alkaline by itself with the pH of 13–14. It is an activator which is also an oxide carrier for more efficiency.

According to Bragg's law, X-ray diffraction analysis entails transmitting X-rays to a sediment sample while also placing a detector around the sample to assess the intensity of the X-rays according to its direction (NF EN 13925-1, 2003). The Nitron XL5+ apparatus was used for XRD measurements. Non-destructive testing X-ray diffraction (XRD) was performed on FA, GGBS, and NeoliX® following the French standard EN 13925-1, 2003, and the findings demonstrate that, like GGBS, FA also contains compounds of calcium, magnesium, aluminum, silicates, as well as of iron and potassium (Table 6).

Table 6. Chemical properties of GGBS, FA, and NeoliX®.

Material	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	MgO	H_2O
GGBS	37.00	10.80	-	-	43.00	7.10	-
FA	46.81	20.45	7.09	4.35	12.52	3.12	-
NeoliX®	36.1	-	-	-	-	-	50%

2.3. Traditional Hydraulic Binders

Ordinary Portland cement (commercial name CEM-I 52.5) results from the grinding of clinker and calcium sulphate (gypsum or anhydrite) to make the mixture, and possibly of secondary constituents in small quantities (less than 5%). The clinker content is at least 95%. It has a resistance to current compression at 28 days of 50 MPa (NF EN 197-1, 2012). OPC and CSA cement for this project were provided by Lafarge cement France. The commercial name of CSA is Neutracem. CSA cement is a mixture of sulfo-aluminous clinker and gypsum or anhydrite to activate its setting.

Calcium oxide, commonly known as lime, is thought to be very effective against highly organic sedimentation. In this investigation, industrial lime was used to remediate organic sediments. Quarry sand can improve the sediment-binder mixture's granular skeleton. Quarry sands fill in the spaces and raise the compactness of the sediment-binder mixture, improving the mechanical characteristics.

The particle or grain size of sediments is a fundamental feature or physical property [62]. Several sediment or material properties, such as texture and appearance, density, porosity, and permeability, are directly influenced by the size of its constituents, as well as their shape. Particle size distribution is one of the most important parameters that control the mechanical strength development. Binici et al. [63] verified that specimens with a better fineness and a narrower particle size distribution had the highest compressive strength. The particle size distributions were measured with LASER granulometry as per standard ISO 13320-1, 2003 (Figure 3). This technique allows the sediments to be classified in relation

to the size of their particles. It is based on the principle of Fraunhofer diffraction: particle rays are measured using a laser beam on sediment particles suspended in water.

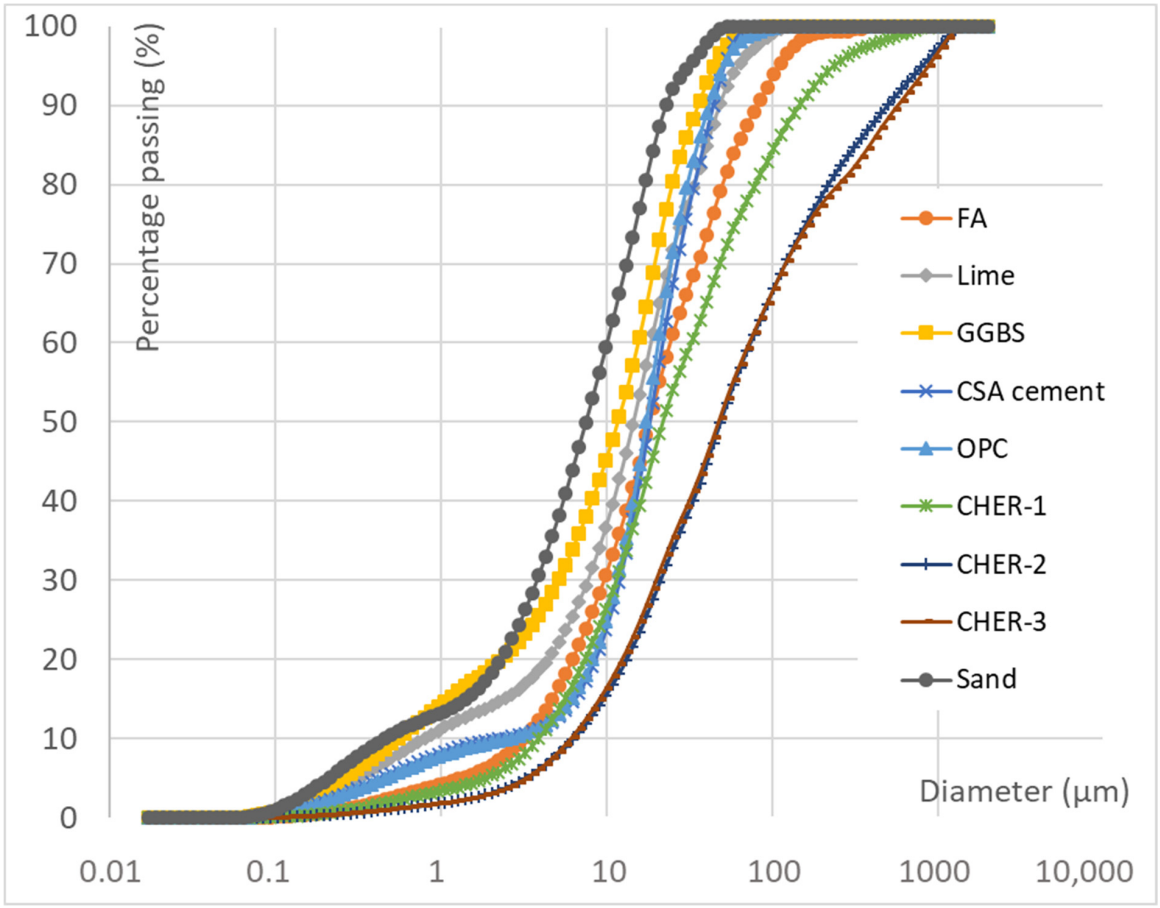


Figure 3. Raw material particle size distribution curves.

The size distribution parameters of the raw materials used in this study were deduced from the particle size distribution curves and are presented in Table 7.

Table 7. Grain size distribution parameters for raw materials.

Material	d10 (µm)	d50 (µm)	d90 (µm)	SSA (cm^2/g)
Lime	0.78	14.39	47.67	3213.70
OPC	2.55	17.18	41.22	2326.10
GGBS	0.54	11.58	35.39	4005.80
FA	1.95	24.66	229.00	1811.50
CSA cement	2.20	18.08	43.68	2396.90
CHER-1	3.88	22.20	152.50	1152.85
CHER-2	6.25	48.39	494.62	688.44
CHER-3	6.03	47.03	565.40	705.30
Sand	0.45	7.45	22.68	4639.80

2.4. Methodology

Mandatory characteristics of the sediments are now well-known, which is the first step in the valorization process [64]. Laboratory-scale experimentation can be commenced once

the choice of binder is made. If laboratory-scale studies yield promising results from both a technical and economical point of view, pilot-scale testing is performed in the next stage (pilot-scale studies are not part of this paper). Otherwise, the laboratory-scale experiments are repeated with different binders with increased dosage or with pre-treatment of sediment prior to the addition of eco-binders. Figure 4 presents the strategy for reusing the sediments.

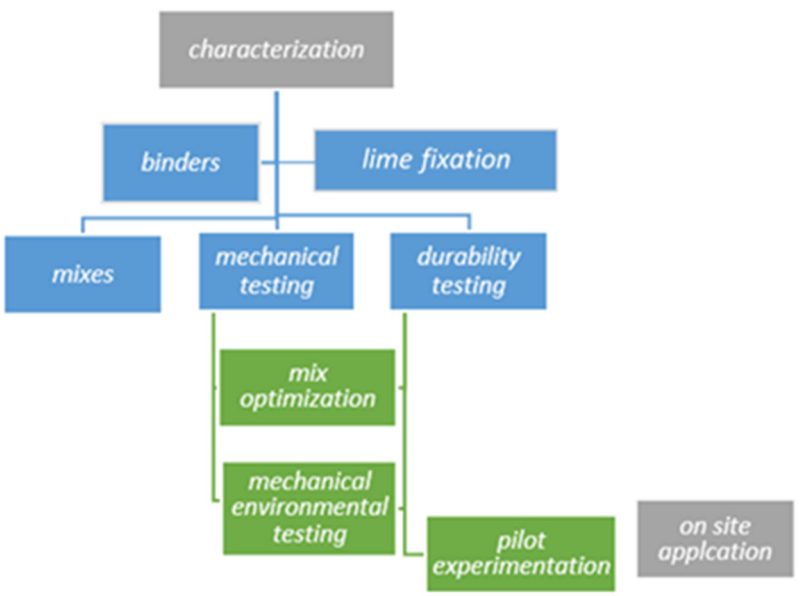


Figure 4. Work flowchart and decision-making steps.

2.4.1. Binder Selection

The choices of binders, depending upon their impacts on the environment, were the following, as shown in Figure 5:

- Low-carbon binders, e.g., eco-binder NeoliX® (used as an activator with a mineral charge).
- Pozzolanic by-product as FA (sodeline).
- GGBS binders, e.g., Ecocem (an ecofriendly solution).
- Specific binders, e.g., Neutracem or CSA cement (with high clinkers).
- Ordinary Portland cement (OPC) (cement mostly consisted of clinker) with or without the addition of lime, depending upon the percentage of organic matter present in the sediments.

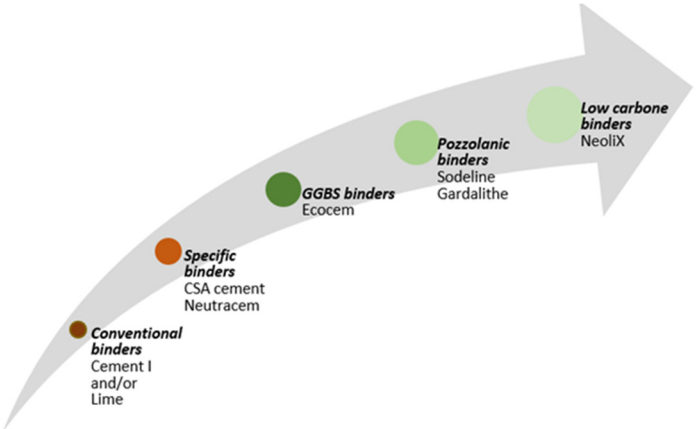


Figure 5. Selection of binders: from standard binders to low-carbon and eco-binders.

2.4.2. Lime Fixation

To determine the percentage of lime needed, the lime fixation test was used. This test consists of measuring the pH of a lime-sediment-water mixture (NF ISO 10390, 2005) and determining for which lime dosage the pH of the suspension reaches a value of more than 11, favorable to the development of cement hydrates [54]. GTR also recommends the percentage of hydraulic binder with respect to the percentage of lime. Lime can be used alone or with other binders, due to its alkaline effect, to tackle organic matter. Lime fixation for CHER-1 sediments reaches high constant pH at around 4.5% of lime, for CHER-2 at around 4.45%, and for CHER-3 at around 4.8% (Figure 6). CHER-1, CHER-2, and CHER-3 contain 16.75%, 15.68%, and 19.68% of organic matter, respectively.

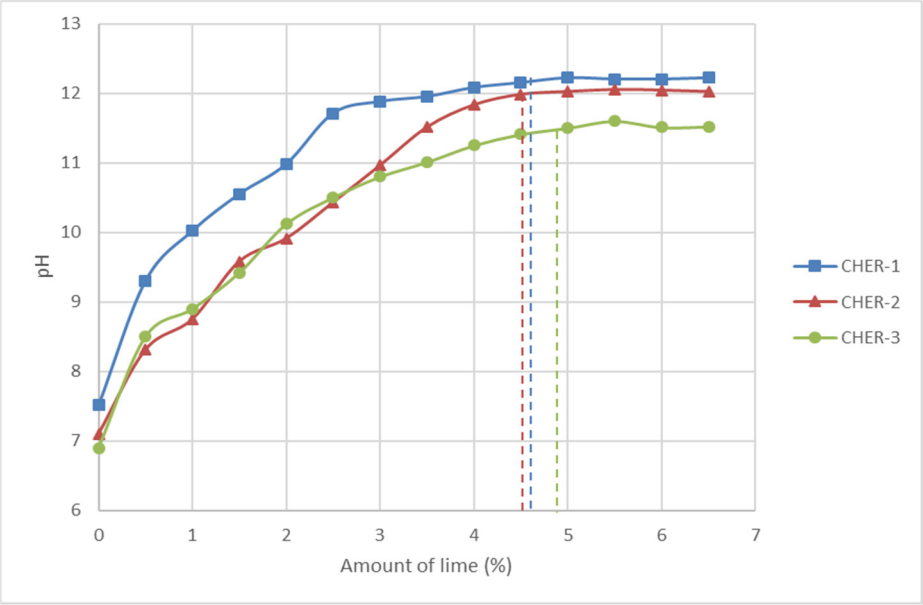


Figure 6. Lime fixation test results.

2.4.3. Sample Preparation

After the characterization of sediments, the preparation of samples through compaction at normal Proctor energy (Proctor test) is the next step before mechanical testing. CHER-1, CHER-2, and CHER-3 were mixed together and named as CHER-ALL, and all the following testing was performed on CHER-ALL. According to the GTR [54], class A1 and A2 materials with low organic matter (OM) contents (OM < 3%) are considered to be good earth-moving materials and can be treated with hydraulic binders to use them as backfill material or for under-layers of roads. Otherwise (OM $\geq$ 3%), the materials must be the subject of a specific study to evaluate their behavior. The average organic matter content in CHER-ALL is 17.37%, which is very high, and dredged sediments will be treated with lime and introduced with QS before introducing them into traditional binders. Eight types of different formulations were considered, as shown in Table 8 from M0 to M7, for traditional hydraulic binders.

Two formulations (M8 and M9) were prepared using pozzolanic binders (FA and GGBS) with the introduction of an activator (Table 9).

Table 8. Eight different formulations under study using traditional binders.

Formation Name	Binder Used	Percentage by Weight (%)	OMC (%)
M0	non	0	29.30
M1	OPC	9	30.5
M2	OPC + lime	6 + 3	30.20
M3	OPC + lime	4 + 5	30.30
M4	OPC + lime + QS	6 + 3 + 25	30.10
M5	OPC + lime + QS	6 + 3 + 15	30.40
M6	CSA cement	9	30.05
M7	OPC + FA	9 + 2	30.10

Table 9. Two different formulations under study using pozzolanic binders.

Formation Name	Binder Used	Percentage by Weight (%)	w (%)
M8	GGBS + activator	Different percentages	50.00
M9	Fly-ash + activator	Different percentages	50.00

In accordance with the requirements of French standard NF P 98 114 3, 2003, relating to the study of materials treated with hydraulic or pozzolanic binders, the diametral compression strength measurement test must be carried out on cylindrical specimens of 40 mm in diameter and 80 mm in length. Samples using hydraulic binders were prepared as shown in Figure 7 by compacting them at normal Proctor energy (600 kN $\times$ m/m^3) as per standard NF P94-093, providing optimum water content.



Figure 7. Sample preparation by compacting them at normal Proctor energy.

A total of 24 samples were prepared for each formulation to measure unconfined compressive strength (UCS) at 7, 14, 28, 60, 90, and 180 days, as well as to test the durability of these mixtures (Table 10). Three samples were tested at each date and the average value was considered.

Table 10. Number of samples for each formulation.

Testing Type	Total Samples
UCS to be tested at 7, 14, 28, 60, 90, and 180 days (3 samples per day)	18
Wet/dry method (w/d), freeze/thaw method (f/th) (3 samples per test)	6

3. Results and Discussion

3.1. Valorization Using Hydraulic Binder

In this section, the results from unconfined compression strength (UCS) and California bearing ratio (CBR) testing are noted for all different types of formulations, from M0 to M7. Increasing axial force was applied on the sample while measuring the displacement until the fracture in the sample occurred (Figure 8). The press-machine itself notes down the final force, and the force per unit area yields the UCS.



Figure 8. Unconfined compressive strength testing.

The unconfined compressive strength (UCS) testing results at 7, 14, 28, 60, 90, and 180 days for each formulation from M0 to M7 are presented in the Figure 9. A slight increase in early days' (7, 14, 28) strength was observed by replacing 3% of OPC with lime (M2 formulation), and by replacing 5% of OPC with lime, the UCS increased by 10–15% at each day (M3 formulation). This fact could be explained with the knowledge about the high organic matter content in the CHER-ALL sediments, and lime has proven to be efficient in neutralizing the organic matter content [65–67].

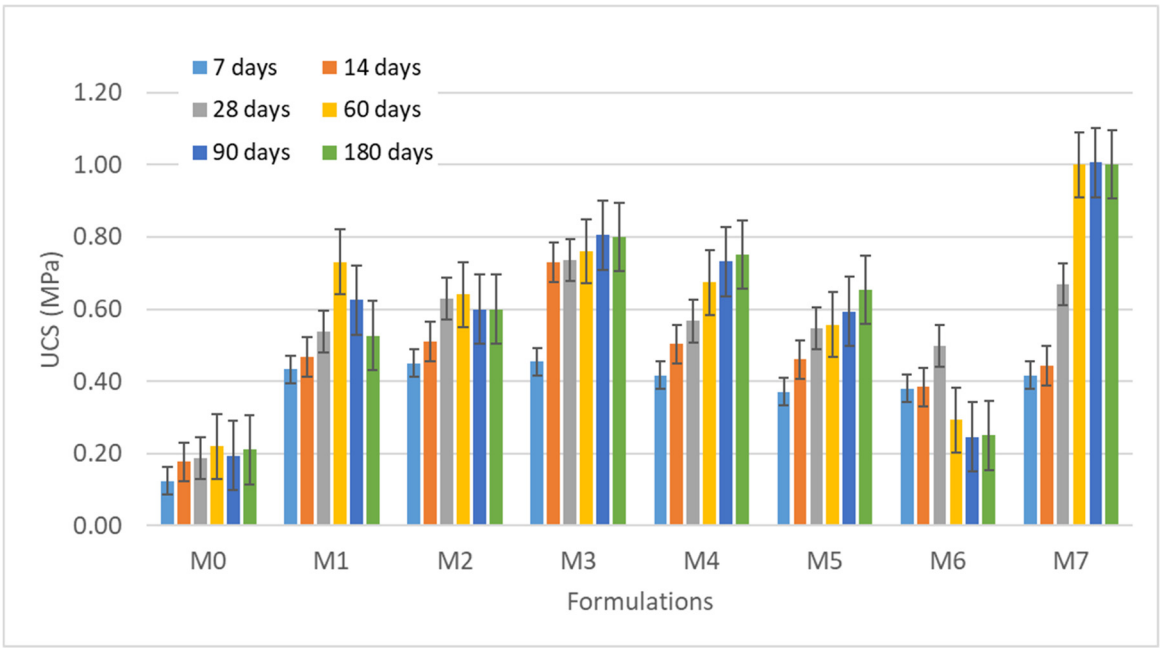


Figure 9. UCS test results at 7, 14, 28, 60, 90, and 180 days for formulations M0 to M7.

The addition of 25% and 15% sand to the M4 and M5 formulations, respectively, did not yield effective outcomes. Except for the M7 formulation, which had a USC value of 1 MPa at 60 days, each formulation had UCS values below 1 MPa at each day. Nine percent OPC and two percent fly ash were added in the M7 formulation to treat the dredged sediment. To determine the durability of combinations against environmental changes, three samples from each formulation were subjected to 20 cycles of freezing and thawing, as well as 20 cycles of wetting and drying (each cycle lasting 24 h). These tests were carried out in the M2C laboratory according to accelerated damage protocols. Every cycle began on the 8th day after preparation and ended on the 28th day, and the findings after 28 days for each formulation are shown in Figure 10.

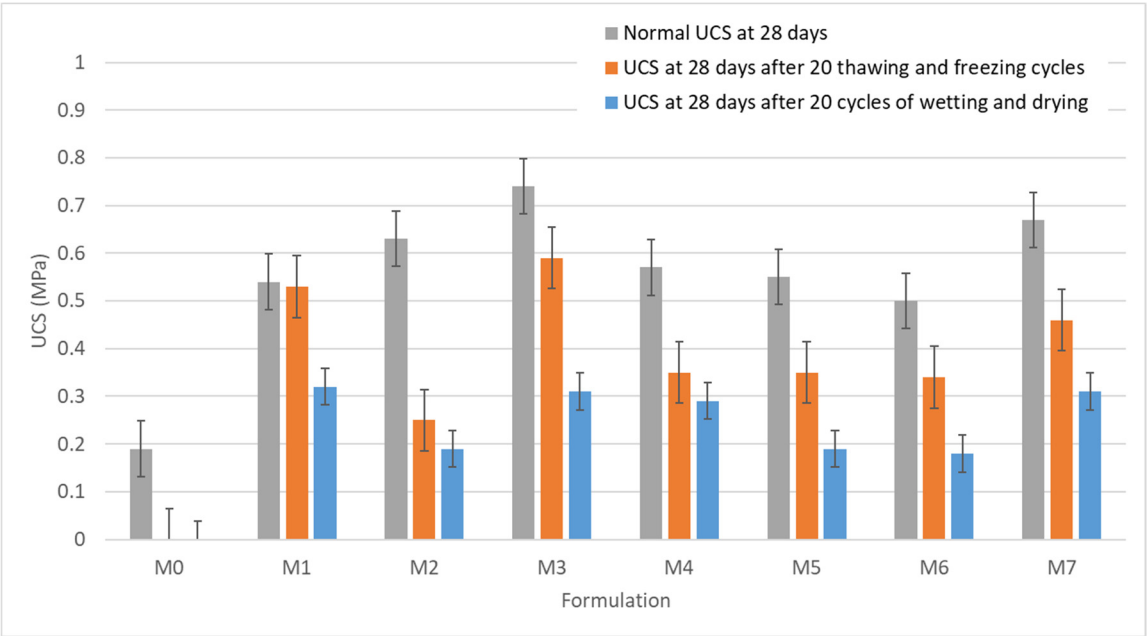


Figure 10. Durability testing results at 28 days for formulations M0 to M7.

After 28 days, the UCS for freezing and thawing, as well as wetting and drying processes, decreased, implying that the formulations are not very stable under environmental degradation. In comparison to disposal, dredged port sediments are increasingly being used as backfill and in other construction applications to save money and the environment. However, the sustainability of these soft organic materials as backfill or in other construction applications is always unclear [67]. The M2 and M3 formulations appear to be suitable for use as backfill materials with these highly organic sediments from the port of Cherbourg.

To use dredged materials for road construction, it is necessary to understand the specific requirements defined by the federal or local authorities of that area. As per French standards, a minimum and a maximum compressive strength (UCS) are recommended: a minimum of 2 to 4 MPa to ensure the bearing capacity of vehicles and a maximum $\leq$ 8 MPa to ensure their re-excavation for the base layer, while for the foundation and sub-base layer, UCS should be $\geq$1 MPa. To use the dredged sediments for road construction, a minimum unconfined compressive strength of 2 MPa is needed for the base layer and 1 MPa for the sub-base layer. Other than that, Proctor and tensile strength tests are also recommended by French rules and regulations for road construction. To obtain the required strengths, the sediments must be treated with a high proportion of hydraulic binder. The Proctor test evaluates the compaction properties of dredged material (European Standard, NF EN 13286-2, 2005). Immediate bearing capacity (IPI or CBR), which determines the capacity of a material to support the circulation of the building machines under construction, is also recommended (European Standard, NF EN 13286-47, 2003).

The recommended characteristics for the different road layers as specified in French standards [68] are presented in Figure 11.

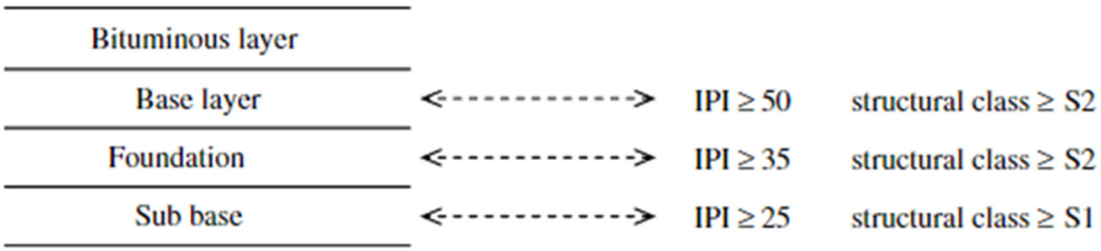


Figure 11. Scheme of road section and recommended engineering characteristics [68].

California bearing ratio (CBR) compares the bearing capacity of compacted material with reference to standard crushed material. As per the French standard (NF P 98-115, 2009), the CBR test was used to evaluate the strength of the subgrade material. Figure 12 presents the CBR testing results for dredged sediments treated with hydraulic binders.

With respect to UCS and CBR values, the results (Figure 12) showed that no formulation from M0 to M6 can be used in road building, but M7 can. The challenge is explained by the fact that the CHER-ALL contains more than 17% OM content. The M2 and M3 formulations appear to be adequate for use as backfill material. Lime is efficient against high OM content (formulations M2 and M3), and the addition of 2% FA to formulation M7, together with 9% of OPC, enhanced the mechanical qualities, making the formulation M7 appropriate for backfill material as well as the sub-base layer of roads.

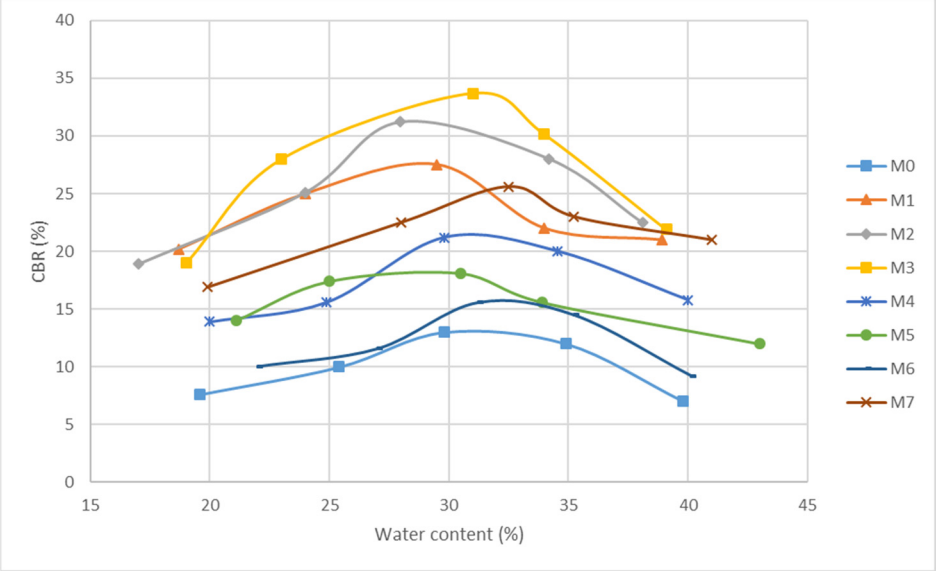


Figure 12. CBR values for each formulation.

3.2. Valorization Using Pozzolanic Binder (Co-Valorization)

The dredged sediments treated solely with GGBS and FA did not produce competitive results. The concept of co-valorization was used in formulations M8 and M9 with the addition of an activator in relation to GGBS and FA to treat the dredged sediments. To understand and define the economical percentage of activator required, initial testing was carried out using only GGBS and FA, incorporating the activator without dredged sediments. For GGBS, no reaction was noticed until 4% of activator, and then a sharp increase was recorded at 9%. On the other hand, FA do not yield competitive results incorporating the activator (Table 11).

Table 11. Introduction of an activator along with GGBS and FA to make a geopolymer.

Activator (%)	Water (%)	GGBS (%)	UCS_{GGBS} (MPa)	FA (%)	UCS_{FA} (MPa)
1	50	99	0	99	0
2	50	98	0	98	0
3	50	97	0	97	0
4	50	96	0	96	0
5	50	95	0.19	95	0
6	50	94	0.2	94	0.12
7	50	93	0.35	93	0.12
8	50	92	1.3	92	0.13
9	50	91	7.1	91	0.136
10	50	90	10.85	90	0.25
15	50	85	14.1	85	0.32
20	50	80	14.9	80	0.56
30	50	70	14.94	70	0.32
40	50	60	8.5	60	0.30
50	50	50	7.03	50	0.25

In contrast to the treatment with hydraulic binders, sample preparation with the geopolymer did not involve compaction. Geopolymers require a lot of water to react chemically, and after a number of tests with different water contents, 50% water content was chosen for further research since it produced the best results. The water serves two purposes: It improves the material's workability, which would be impossible without it because the activator is a type of prepolymer that is exceedingly viscous. It is difficult to make a usable liquid activator that can react without this water. Additionally, the added water generates a large enough reaction media for the reaction to occur. Karam et al. [31] explain that an increase of the water content affects the mechanical behavior of the binder (alkaline-activated GGBS) more than sediments' incorporation. Water content is directly linked to the mechanical behavior of the geopolymer, and therefore optimization was necessary, and the final water to solid mass (W/S) ratio was 0.50.

Since the FA-activator combination was unsuccessful, co-valorization of CHER-ALL utilizing FA-geopolymer activation appears to be impossible. GGBS, on the other hand, reacted efficiently with the activator, making it a good candidate for co-valorization. The silicates provided by the activator combine with the calcium cations yielded by the GGBS dissolution to produce dense C-A-S-H structures, providing a better binder strength [69]. Unlike FA, GGBS contains a high percentage of CaO, and therefore, yields a high number of calcium cations upon dissolution.

Keeping in mind the strength as well as economical parameters, the percentage of activator selected for future studies was 10%. With 10% of activator, 90% of GGBS, and 50% of water by weight, the UCS obtained was more than 10 MPa. The dosage of activator and percentage of water were kept constant. The percentage of GGBS was replaced step-by-step with the Cherbourg sediments (CHER-ALL) by keeping the W/S ratio as 0.50, as shown in Table 12.

Table 12. Replacement of GGBS with dredged sediments.

Water (%)	Activator (%)	GGBS (%)	Sediments (%)	Reference
50	10	80	10	CHN10
50	10	70	20	CHN20
50	10	60	30	CHN30
50	10	50	40	CHN40
50	10	40	50	CHN50

The unconfined compressive strength testing was performed for each formulation at 7, 14, 28, 60, 90, and 180 days. Figure 13 shows the UCS development at each day. It can be seen that the UCS for CHN30 was 2 MPa at 28 days, and higher at 60, 90, and 180 days, which is suitable to be used as a road construction material. Replacing 40% and 50% of GGBS with CHER-ALL has reduced the UCS below 1 MPa and 0.3% at 28 days, respectively.

To further improve the formulation CHN30, the introduction of lime and granicalcium® (granular calcium carbonate 0.34/0.7 mm, from Omya, France) was performed, as shown in Table 13.

Table 13. Replacing 3% and 5% of GGBS with granicalcium and lime, respectively, in formulation CHN30.

Water (%)	NeoliX® (%)	GGBS (%)	Sediments (%)	Lime (%)	Granicalcium (%)	Reference
50	10	60	30	0	0	CHN30
50	10	55	30	5	0	CHN30L
50	10	57	30	0	3	CHN30C

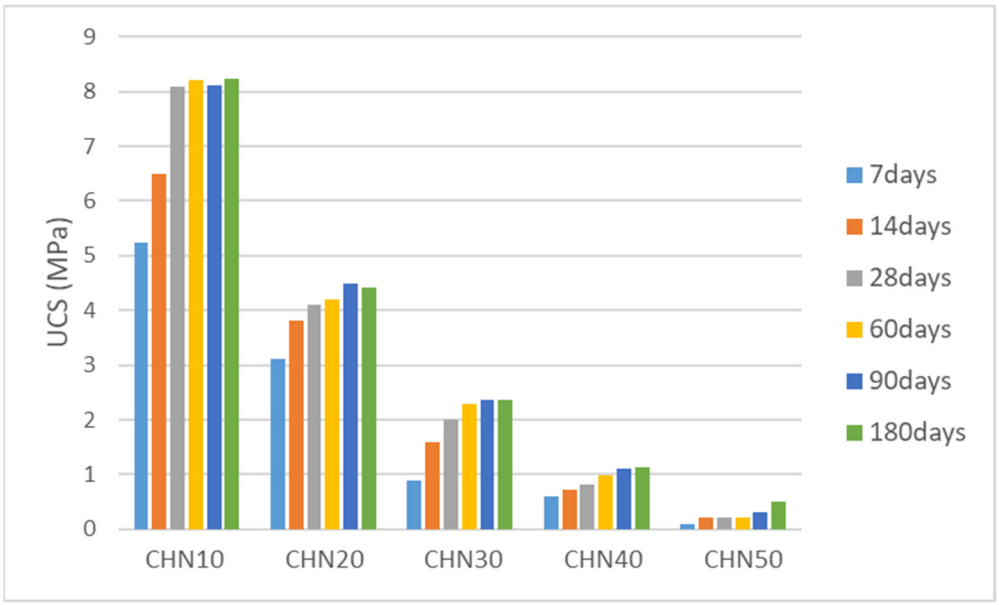


Figure 13. UCS (MPa) testing results for CHER-ALL treated with a geopolymer.

UCS and tensile strength (Rt) tests were performed at 7, 14, 28, 60, 90, and 180 days after the addition of lime and granicalcium to the CHN30 formulation (Figure 14).

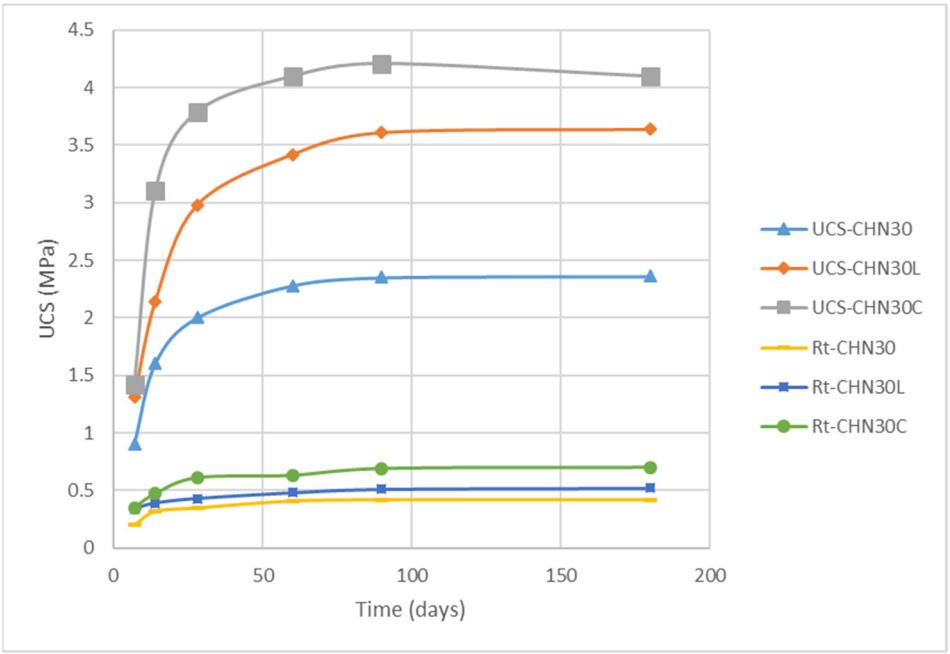


Figure 14. UCS study of CHN30 with the introduction of lime and granicalcium.

After dissolution, the addition of lime and granular calcium carbonate increased the amount of calcium cations available. As previously stated, when the activator's silicates interact with the calcium cations produced by the GGBS dissolution, dense C-A-S-H structures are formed, giving the binders more strength. As a result, the addition of lime and granular calcium carbonate significantly increased the UCS and Rt. The addition of 5% lime in the CHN-30 formulation increased the UCS values by 50% to 60%, and tensile strength by 20% to 25% at each day, and the addition of 3% of granular calcium carbonate increased the UCS by 80% to 90%, and tensile strength by 25% to 30% at each day.

4. Conclusions

The present study reported an analysis of comparative results from two series of tests on treated sediments. Treatments concerned the valorization of highly organic marine sediments in filling material, backfill, or road layers using conventional binders and alkali-activated industrial by-products. The first series of testing included hydraulic binder-based mixtures. The second series investigated the possible use of GGBS as a precursor for developing a geo-composite material with the same sediment. In this series, at first, the addition of 10–50% of dredged sediments was tested, followed by optimization using lime and granular calcium carbonate. The analyses performed on the mechanical properties of each mixture and on the two series were as follows:

Concerning hydraulic binder-based mixtures, i.e., the first series:

- The UCS values obtained ranged from 0.12 to 0.21 MPa for M1, 0.43 to 0.60 MPa for M2, 0.45 to 0.80 MPa for M3, 0.42 to 0.75 MPa for M4, 0.27 to 0.65 MPa for M5, and 0.38 to 0.50 MPa for M6. Similarly, the CBR values were below 35% for each formulation from M0 to M6. These values did not match with the required values for road sublayers' application according to the French road engineering guide, GTR (2000).
- Formulation M7, which included 9% OPC and 2% FA, had the maximum UCS (1 MPa) after 60 days, with a maximum tensile strength of 0.14 MPa at 28 days, and the maximum value of CBR was 33.68. The estimated value of tensile strength at 360 days for materials treated with OPC can be calculated from measured values at 28 days as per Equation (1) according to the French standard NF EN 14227-1, 2013:

$$\frac{\mathbf{R_{t28}}}{\mathbf{R_{t360}}} = \mathbf{0.60} \quad (1)$$

Similarly, the estimated values at 360 days for Young's modulus (E) can be calculated from measured values at 28 days according to Equation (2) following French standard NF EN 13286-41, 2003:

$$\frac{\mathbf{E_{28}}}{\mathbf{E_{360}}} = \mathbf{0.65} \quad (2)$$

French standard NF EN 14227-1, 2013, suggests the classification of mixtures according to their performance values (Rt, E) at 360 days (Figure 15).

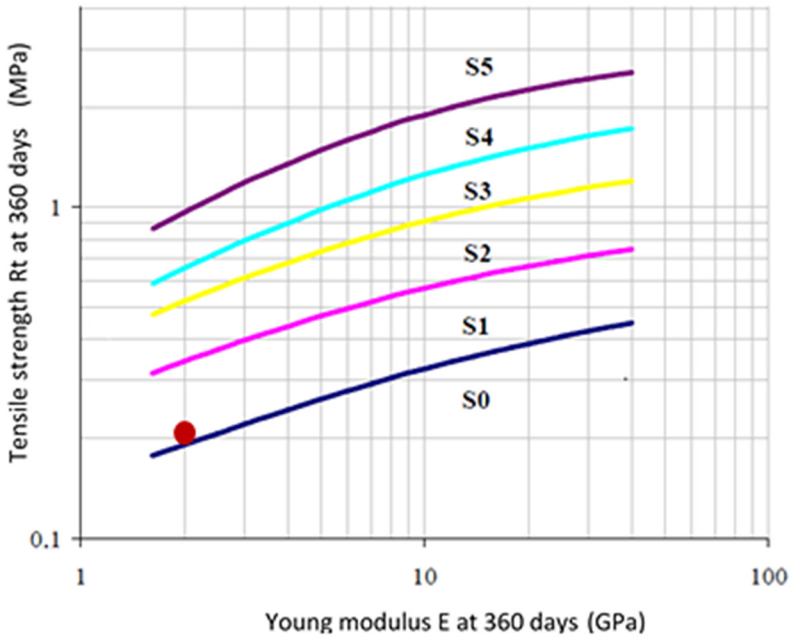


Figure 15. Mixture classification as per French standard.

The M7 formulation barely fulfilled the French criteria to be used in the sub-base layer of roads as it had a UCS value of 1 MPa at 28 days, CBR above 30%, and mixture class S1, as shown by the red dot in Figure 15.

- The addition of lime had a positive effect on strength development in formulations M3 and M4: more lime, higher UCS. Lime was used against OM: increases were seen in UCS because lime plays a role on the OM activity and decreased its inhibition against hydration.
- The addition of a granular corrector did not yield positive results, which could be due to the high sand content (>25%) already present in the dredged sediments.
- The relatively low-carbon print CSA cement, as compared to OPC, did not show promising results. Like OPC, CSA cement also had difficulty in stabilizing these sediments due to the presence of a high OM content (>17%).

Before the second series of tests (with geopolymer), preliminary tests (Table 11) were conducted using GGBS and FA along with an activator only. This allowed to observe the strength development with GGBS and FA when mixed with the activator and to select the optimal percentage of activator (10%).

Concerning the geo-composites with GGBS mixtures and activator, i.e., the second series:

- After a series of testing, the optimal water to solid mass ratio (W/S ratio) was decided to be 0.50, as below this ratio, mechanical strength values significantly decrease. A panel of 5 tests were performed, that included 10% to 50% of dredged sediments to observe the strength development. The influence of the addition of sediments in the silicate-activated system revealed that the UCS value had decreased. At 30% by weight of dredged sediments' addition to the geopolymer (activator + GGBS), the UCS value obtained was above 2 MPa at 28, 60, 90, and 180 days.
- Three tests were carried out on the addition of calcium, either with lime or granular calcium carbonate (granicalcium). A significant increase in UCS was observed, where the addition of 5% lime increased the UCS up to 60% and Rt up to 25%, while the addition of 3% granular calcium carbonate increased the UCS up to 90% and Rt up to 30%.
- With the UCS and Rt levels obtained, it will be possible to consider these geo-composites as road sublayer materials with adapted processes in practice.

These series of tests demonstrated that conventional binders (binder dosages < 10%) are not able to develop significant and required strength performance with highly organic sediments (>17%) for road layers' applications. As filling materials, it would be possible after considering the compaction abilities.

However, alkali-activated by-products such as GGBS could constitute, with a significant quantity of highly organic sediments, a promising geo-composite able to be reused in road and geotechnical engineering.

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Conflicts of Interest: The authors have no conflict of interest.

Future Work Recommendations: The future works should focus on the optimization of geo-composite materials (sediments-GGBS-NeoliX-activator) considering highly organic sediments, and also water contained by the dredged sediments to minimize the dewatering for valorization. As observed by other testing, the water/(GGBS + sediment) or W/S ratio influences the UCS, and certainly the workability and setting time of mixtures, and it will be necessary to conduct further tests on the W/S influences and to optimize the formulation for a maximal mass incorporation of sediments.

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