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Alkaline activation of mussel shells as an innovative binder for geotechnical soil improvement

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ABSTRACT: The increasing availability of waste and industrial by-products has driven researchers to explore their potential in developing sustainable construction materials. Among these, alkali-activated binders (AABs) have emerged as a promising eco-friendly alternative to traditional binders like lime and Ordinary Portland Cement (OPC) as additive in soils improvement, offering both environmental and performance advantages. This study investigates the use of mussel shell powder (MSP), rich in calcium carbonate ($CaCO_3$), as a precursor for synthesizing alkali-activated binders (AAB). AABs are known for their ability to lower CO_2 emissions and their potential to incorporate industrial and natural by-products, such as pozzolans, blast furnace slags, and waste limestone. However, while research has primarily focused on aluminosilicate-rich precursors, the potential of $CaCO_3$-based materials remains underexplored. This study evaluates the effectiveness of MSP combined with two types of alkaline solutions of different chemical composition: i) 12 M sodium hydroxide (NaOH) solution and ii) a mixtures of 12 M NaOH and sodium silicate (Na_2SiO_3) solutions with a fixed SiO_2/Na_2O mass ratio of 1.7. Physical and mechanical behaviour of the binders are analysed by means of Ultrasonic Velocities (UV) measurements and Unconfined Compressive Strength (UCS) tests. The reactivity of the activated MSP is monitored over time using Thermogravimetric analysis (TGA). The mineralogical investigations are fundamental to support the interpretation of the mechanical performance of the binders. The results demonstrate that calcium carbonate dissolution generates metastable gels that crystallize into hydrated carbonates, such as pirssonite and gaylussite. The availability of silicon further promotes pozzolanic reactions and polycondensation processes, resulting in the formation of hydrated silicate gels. These chemical transformations enhance the mechanical performance of the binders, confirming the potential of MSP as an innovative and sustainable component in alkali-activated binders (AABs). An insight into the mechanical effects induced by AAB based on MSP on a dredged marine sediment is provided, highlighting the role of silicon availability on the effectiveness of the treatment.

KEYWORDS: Mussel shell powder, alkali-activated binders, waste material, soil, sustainable material.

1 INTRODUCTION

Using seashells as a biogenic precursor for the synthesis of novel binders, an alternative to lime and Portland cement, could offer a promising option for specific geotechnical applications, such as soil stabilization. Reusing biogenic $CaCO_3$ as an alkali-activated binder is particularly practical because it is environmentally friendly and cost-effective. This approach reduces the carbon footprint since it avoids using primary resources, and calcination is not required for activation, leveraging the abundant, low-cost availability of these resources.

Mussel shells are a widespread food waste from aquaculture industry. Their global production for human consumption is more than 15 million tonnes per year (Wijsman et al. 2019), which is about 14% of the total marine production. Rich in calcium carbonate (95%), shells are a non-biodegradable type of waste, which is difficult to compost and requires long and expensive disposal (Morris et al. 2019).

Attempts in looking for viable and sustainable alternative for the valorization of these by-products are ongoing in different fields of application. Several studies have been addressed to the reuse of seashell wastes for concrete production as a partial and total coarse aggregate replacement, filler and cement replacement, in crushed, ground or powdered form (Bamigboye et al. 2021; Cuadrado-Rica et al. 2015; Kuo et al. 2013; Nguyen et al. 2013; Ishak et al., 2021).

Martinez-Garcia et al. (2017) evaluated the effect of replacing fine and coarse aggregates with mussel shells in mortar mixtures. Razali et al. (2017) investigated the partial substitution of ordinary Portland cement with calcined mussel shell powder (MSP), highlighting that its effectiveness is limited to low replacement ratios, as higher dosages result in poor bonding within the concrete matrix. Stel'makh et al. (2022) identified the optimal dosage of MSP for producing modified concrete with enhanced properties. Petti et al. (2024) examined the influence of MSP on the mechanical behaviour of shell-cement mixtures when used as a partial cement replacement for the geotechnical improvement of dredged sediments.

New research trends concern the use of $CaCO_3$-rich materials as partial precursor replacement also in alkali-activated alumino-silicate binders, which are generally produced by making react calcined clays (i.e., metakaolin) or industrial waste (i.e., coal fly ash or steel slags) with an alkaline solution (Davidovits, 1991; Palomo, 1999; Duxson, 2007). A literature review on the effect of limestone on the engineering properties of alkali activated binders synthesised from various precursors (i.e., fly ash, slag, metakaolin, and blended systems) was provided by Chan et al. (2023) focusing on the reaction kinetics, microstructure, fresh and hardened properties and sustainability. Recent literature focuses on the use of calcium carbonate minerals as the only precursor in the synthesis of AABs.

The alkaline activation of $CaCO_3$-rich materials is a chemical process driven by an activator (i.e., an alkaline solution containing hydroxides and/or silicates) that promotes the dissolution of the calcium carbonate source and the subsequent formation of metastable and crystalline carbonate phases such as pirssonite ($Na_2Ca(CO_3)_2{\cdot}2H_2O$), gaylussite ($Na_2Ca(CO_3)_2{\cdot}$

$5H_2O$), portlandite ($Ca(OH)_2$) and thermonatrite ($Na_2CO_3 \cdot H2O$). These phases can be finely intermixed with amorphous calcium silicate hydrates (CSH), silica gel, sodium-containing calcium-silicate hydrates (NCSH), and/or sodium silicate hydrates (NSH), depending on the activator type used (Avila-Lopez et al. 2015; Ortega-Zavala et al. 2019). The reaction can occur at ambient temperature and pressure, and its extent strongly depends on formulation parameters, such as the initial $CaCO_3$ ratio and the activator type, which, in turn, influence the workability and mechanical strength of the binder (Hanein et al. 2021; Cousture et al. 2021). Ortega-Zavala et al. (2019) reported that pastes and mortars produced by activating limestone with sodium hydroxide and sodium silicate solutions reached compressive strengths of 15-25 MPa after 360 days. Similarly, Firdous et al. (2021) investigated the reactivity of natural and synthetic calcium carbonate minerals in sodium silicate solutions. Cousture et al. (2021), in their study on the alkaline activation of limestone using aqueous sodium hydroxide solutions (i.e., 12 M and 13 M) without the addition of active silicate phases, demonstrated that the compressive strength of mortars is strongly influenced by formulation parameters, namely the alkaline solution-to-limestone ratio (AS/L) and the molarity of the NaOH solution. Regarding seashell waste as a source of calcium carbonate minerals, only one study has been reported in the literature, investigating the activation of queen scallop shell powder with a sodium silicate solution (Hasnaoui et al. 2021). However, no previous research has addressed the potential use of bio-based $CaCO_3$ alkali-activated binders for geotechnical applications.

In this study, an experimental investigation is conducted on the mineralogical evolution and mechanical behaviour of AABs produced from MSP. Two alkaline solutions with different chemical compositions are used for activation: a 12 M NaOH solution and a mixture of 12 M NaOH with Na_2SiO_3. The physical and mechanical performance of the binders is assessed through Ultrasonic Velocity (UV) measurements and Unconfined Compressive Strength (UCS) tests, while the reactivity of the activated MSP is monitored over time using Thermogravimetric analysis (TGA). Furthermore, a first attempt is made to evaluate the effects of Alkali Activated Mussel Shells Binders (AAMSB) on the mechanical properties of a dredged marine sediment, based on UCS tests and UV measurements carried out at different curing time (7 and 28 days). The findings provide novel insights into the role of silica species availability in enhancing the effectiveness of the activation process, both in terms of binder performance and the mechanical improvement of treated soils (Vitale et al. 2025).

2 MATERIALS AND METHODS

2.1 *Materials*

2.1.1 *Mussel shells*

The seashell waste used in this study consists of *Mytilus Galloprovincialis* mussel shells sourced from seafood suppliers in the province of Taranto, Italy. These shells are composed of biocrystals and extracellular organic matrices, arranged in distinct structural layers separated by an interface zone. The outer layer is characterized by stacks of fibrous calcite.

MSP is produced through a multi-step process: shells are first washed with tap water for approximately 10 minutes, oven-dried at 105°C for 48 hours to remove residual moisture, impurities, and part of the organic matter (Olufemi et al. 2009; Yang et al. 2005), and then milled and sieved to obtain a particle size distribution with a $D_{50} = 6.32$ µm (Figure 1). The specific surface area is 3.43 m^2/g, and the pH is 8.8. Mineralogical analysis shows that MSP is mainly composed of calcite (73.0 wt %) and aragonite (26.7 wt %), with trace amounts (about 0.3 wt %) of quartz. Chemically, it contains predominantly calcium oxide (CaO = 58.57 wt %), with minor quantities of SiO_2 (0.36 wt %), Na_2O (0.37 wt %), MgO (0.19 wt %), and Fe_2O_3 (0.13 wt %).

Figure 1. a) the mussel shell; b) mussel shell granule; c) mussel shell powder.

2.1.2 *Alkaline solutions*

The alkaline activators used for the synthesis of the AAMSB are a 12 M NaOH solution and a mixture of 12 M NaOH with Na_2SiO_3 solution. The 12 M NaOH solution is prepared by dissolving 480 g of NaOH pellets (99% assays) in 1 L of distilled water, and then stored at ambient temperature for at least 24 h to allow cooling before use. The sodium silicate solution (SS-Na_2SiO_3), supplied by Woellner (Ludwigshafen, Germany) has a SiO_2/Na_2O mass ratio of 1.7. For the preparation of the mixed activator (12 M NaOH + Na_2SiO_3), the weight ratio between NaOH (12 M, aq) and Na_2SiO_3 (aq) solutions is fixed equal 0.5.

2.1.3 *Marine sediment*

The marine sediment was dredged from the Port of Taranto, southern Italy and is classified as a clayey silt with a low sand fraction. It exhibits a high natural water content (w = 80%) and relatively high organic matter content (TOC = 1.52%), consistent with the low specific unit weight of solids ($\gamma_s = 15.95$ kN/m³). The liquid limit (w_L) and plasticity index (PI) are 53% and 28%, respectively, classifying the material as a high-plasticity clay (CH) according to the Unified Soil Classification System (USCS; ASTM D2487-17e1). The consistency index (CI) is -0.96, indicating a fluid state, which is further confirmed by the low undrained shear strength (C_u) measured on undisturbed samples via laboratory vane (LV) tests, with an average value of 7.5 kPa.

2.2 *Samples preparation*

The AAMSBs are prepared by hand-mixing mussel shell powder with the alkaline activators in fixed proportions. The binder produced using 12 M NaOH solution is referred to as AAMSB-12M, whereas that prepared with the combination of 12 M NaOH and Na_2SiO_3 solutions is referred to as AAMSB-12MSS. The alkaline activator-to-mussel shell powder mass ratio is set at 0.6. The fresh AAMSB-12M and AAMSB-12MSS mixtures are cast into polycarbonate moulds, sealed in a plastic bags to avoid air exposure, and cured at room temperature (23 ± 2 °C) for different curing times (7, 28 and 40 days) before performing tests.

For soil treatment, the dredged sediment is mixed with 40% of binders (AAMSB-12M and AAMSB-12SS) by wet weight of the sediment. The reconstituted samples, labelled S+AAMSB-12M and S+AAMSB-12MSS, are prepared by pouring the mixtures into moulds without compaction and cured for 7 and 28 days before testing.

2.3 *Experimental procedures*

The macroscopic behaviour of the binders is assessed through UCS tests and complementary ultrasonic wave velocity measurements. UCS tests are performed on cylindrical specimens using a Wykeham Farrance testing machine, with a maximum load capacity of 500 kN and a displacement rate of 1.00 mm/min. Ultrasonic wave velocities are measured in accordance with UNI EN 14579 using a BOVIAR DSP UTD 1004 ultrasonic device equipped with a pair of 55 kHz transducers in direct arrangement. Adequate acoustic coupling between samples and transducers is ensured by applying a thin layer of water-soluble gel (GIMA, Italy).

The mineralogical composition of the binders is analysed via thermogravimetric analyses (TGA) using a Netsch STA 449 F5 Jupiter coupled a QMS 403 D Aëolos. Specimens are heated from 30 °C to 1000 °C at a constant rate of 10 K/min under an air atmosphere.

3 RESULTS

The mechanical behavior of AAMSB-12M samples, in terms of stress-strain curves after 7 and 28 days of curing, is shown in Figure 2. Two tests were performed on binder samples for each curing time, as indicated in the legend (i.e., 1, 2_7d and 1, 2_28d). AAMSB-12M samples exhibited a slight decrease in mean UCS value with curing time, from 69.5 kPa at 7 days to 64.6 kPa after 28 days. Alongside the reduction in shear strength, these samples also displayed a decrease in stiffness over time, as evidenced by the reduction in ultrasonic velocity (UV= 1017.4 m/s at 7 days and UV= 898.1 m/s at 28 days), in line with the UCS results. After a longer curing period (40 days), samples showed a marked degradation in their physical and mechanical properties, preventing the execution of UCS and UV measurements.

For AAMSB-12MSS samples, the mechanical behaviour in terms of stress-strain curves is presented in Figure 3 for curing times of 7, 28 and 40 days. Two tests were conducted for each curing time, as indicated in legend (i.e., 1, 2_7d; 1, 2_28d; 1, 2_40d). A steady increase in mean UCS value was observed with curing time, indicating a gradual improvement in the binder's mechanical performance: UCS rose from 1000 kPa at 7 days to 1416 kPa at 28 days, with a further slight increase after 40 days. As expected, UV values also increased over time, from 993.3 m/s at 7 days to 1102.4 m/s at 28 days and 1134.4 m/s at 40 days. Figure 4 presents images of AAMSB-12M and AAMSB-12MSS samples after UCS testing. In both cases, a brittle failure mode is observed, characterised by the formation of a shear band. The relationship between UCS and UV for the two binders is reported in Figure 5. Compared to alkali-activated MSP prepared solely with 12 M NaOH solution, AAMSB-12MSS samples exhibit a significant increase in both UCS and UV over curing time, whereas AAMSB-12M binders display the opposite trend.

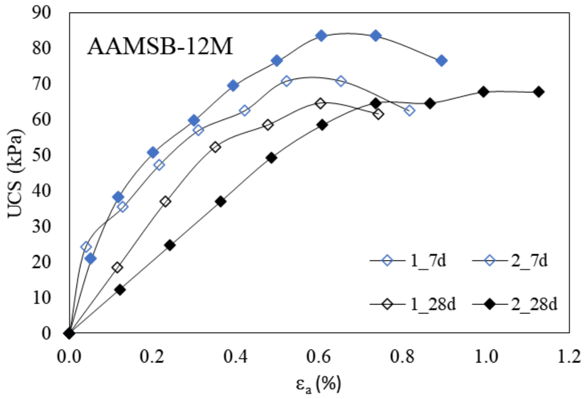

Figure 2. Stress strain curves of AAMSB-12M over curing time.

The mineralogical composition of MSP is altered by the alkaline activation process. The derivative thermogravimetric (DTG) curves of MSP and AAMSB-12M at 7 and 28 days of curing are shown in Figure 6. Both the raw MSP and the alkali-activated samples exhibit their main weight loss between 600 °C and 950 °C, corresponding to the decomposition of calcium carbonate. In the activated samples, this decomposition peak is slightly shifted towards lower temperatures (from 840 °C in MSP to 800 °C in AAMSB-12M-28d), consistent with the presence of alkalis in the mixtures (Avila-López et al. 2015, Misra et al. 1993, Anbalagan et al. 2009). Additional peaks at approximately 125 °C and 175 °C are detected in AAMSB-12M, attributed to the dehydration of gaylussite and pirssonite, respectively.

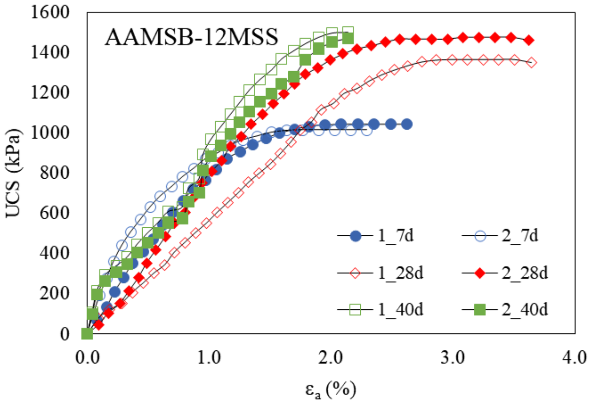

Figure 3. Stress-strain curves of AAMSB-12MSS over curing time.

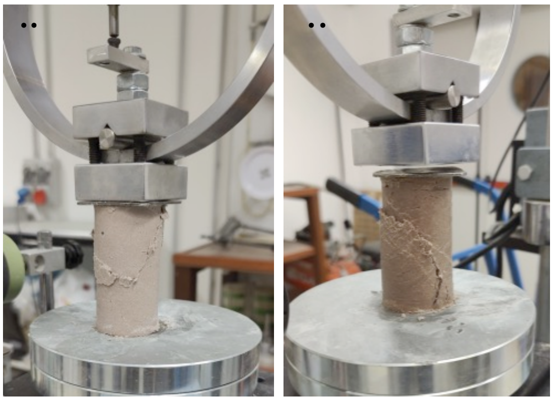

Figure 4. Images of the samples after UCS tests: a) AAMSB-12M; b) AAMSB-12MSS.

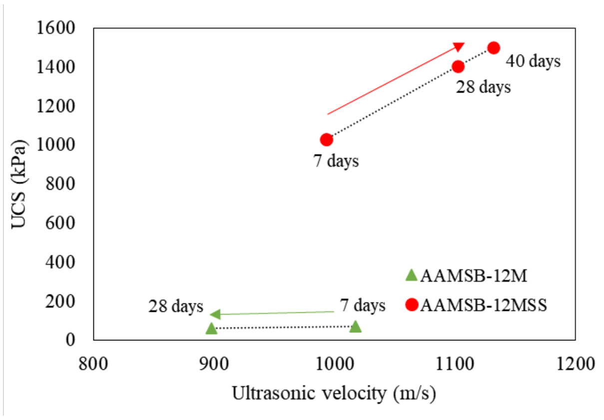

Figure 5. UCS vs. Ultrasonic velocity of AAMSB-12M and AAMSB-12MSS at increasing curing time.

From 7 days of curing onwards, the mass loss associated with gaylussite increases with curing time, while no significant variation in the mass loss associated with pirssonite is observed up to 28 days. For AAMSB-12MSS, the DTG curves at 7 and 28 days of curing are presented in Figure 7. Compared to raw MSP, the alkali-activated sample at 7 days shows a distinct thermal decomposition profile for calcium carbonate in the 600 - 950 °C range, with an initial mass loss between 600 – 700 °C, likely related to carbonate-containing reaction products (Firdous et al. 2021), followed by a second mass loss between 700 – 900 °C corresponding to the decomposition of unreacted $CaCO_3$. This latter peak is shifted towards lower temperatures in the presence of alkalis and newly formed carbonates (Avila-López et al. 2015). Additional peaks at around 125 °C and 175 °C, associated with the dehydration of gaylussite and pirssonite, respectively, are also observed. After 28 days, the AAMSB-12MSS sample shows further $CaCO_3$ decomposition between 700 – 950 °C, an increase in weight loss at 175 °C due to additional pirssonite formation, and no significant change in the mass loss for gaylussite (peak around 125 °C). Moreover, at this curing stage, the 600 – 700 °C mass loss is no longer detected, whereas a new weight loss is observed between 140 - 170 °C (peak around 160 °C), which can be attributed to precipitation/dehydration of new hydrated compound/gels as C-S-H and C-N-S-H. The formation of these phases is consistent with the dissolution of calcium carbonate and the availability of silicon from the alkaline solution (Hasnaoui et al. 2021). Mineralogical analyses highlight the influence of silicon content on the mineralogical evolution of activated mussel shell powder and its consequent effect on the mechanical behaviour of the binder.

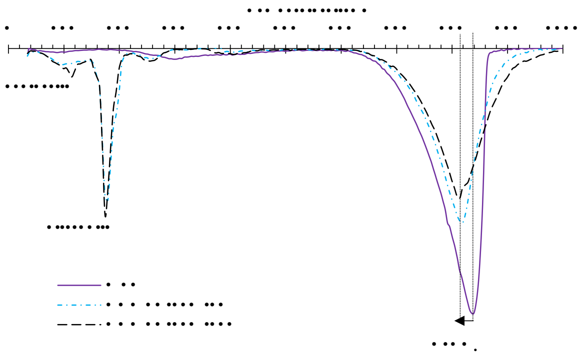

Figure 6. DTG curves of MSP and AAMSB-12M as function of curing time.

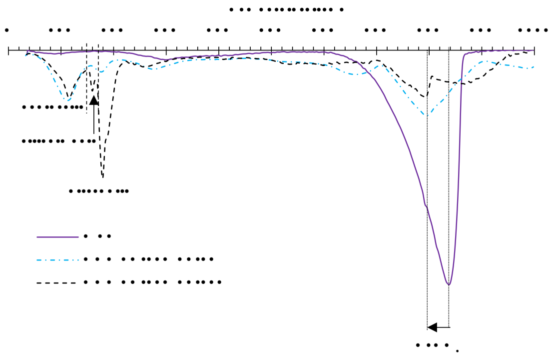

Figure 7. DTG curves of MSP and AAMSB-12MSS as function of curing time.

In AAMSB-12M samples, the dissolution of calcium carbonate in the alkaline environment leads to the precipitation of sodium/calcium carbonate hydrates (i.e., pirssonite and gaylussite) through a recrystallisation process (Sawada 1997). During dissolution and crystallisation, precipitates first form as gels and are subsequently transformed into crystalline products (Brooks et al. 1950). The transformation of metastable carbonate gels into stable crystals reduces stiffness and shear strength, thereby weakening the structure and mechanical performance of the activated MSP. Compared to MSP activated solely with 12 M NaOH solution, the presence of silica species released from the activator in the AAMSB-12MSS binder promotes the precipitation of silicate hydrated gels (Stade 1989; Macphee, 1989), which are responsible for the higher UCS and UV values observed over time.

Figure 8 (a and b) presents the time evolution of UCS and UV values for S+AAMSB-12M and S+AAMSB-12MSS samples. The sediment treated with AAMSB-12M exhibits a UCS of 9 kPa at 7 days and UV of 244.45 m/s, with no significant change over the 28-day curing period. Conversely, sediment treated with AAMSB-12MSS shows a marked increase in UCS, from 53 kPa at 7 days to 74.5 kPa at 28 days. This strength gain is accompanied by an increase in stiffness, as indicated by ultrasonic velocity measurements (UV = 608 m/s at 7 days and UV = 911 m/s at 28 days). These results confirm that the treatment induces changes in the physical and mechanical properties of the dredged sediment, although the extent of improvement and its development over time are strongly influenced by the availability of silica species in the alkaline activator.

4 CONCLUSION

This work presents an experimental investigation on alkali activated binders produced from mussel shell powder as a precursor. The study highlights the role of silica in the mineralogical evolution of the binder and its influence on the macroscopic behaviour of the samples, assessed through the use of two alkaline activators (i.e., 12 M NaOH and 12 M NaOH + Na_2SiO_3 solutions). In both cases, the high-pH environment promotes the dissolution of calcium carbonate and the formation of metastable carbonate gels, which subsequently crystallise into pirssonite and gaylussite.

When silicon is absent from the activating solution (i.e., 12 M NaOH), a progressive decrease in mechanical properties is observed over time, attributable to the transformation of metastable carbonate gels into crystalline phases. In contrast, the presence of silica species in the activating solution (i.e., 12

M NaOH + Na_2SiO_3) promotes the precipitation of silicate-hydrated gels. The enhanced mechanical performance of binders activated with 12 M NaOH + Na_2SiO_3, compared to those produced with NaOH alone, is consistent with the bonding effect provided by stable cementitious compounds such as silicate-hydrated gels.

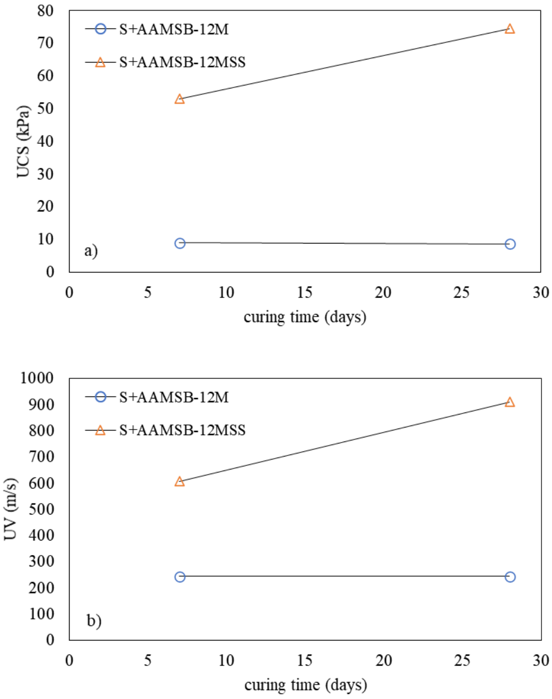

Figure 8. S+AAMSB-12M and S+AAMSB-12MSS samples: a) UCS values over curing time; b) UV values over cutting time.

The study also offers insight into the influence of bio-based alkali-activated binders on the mechanical performance of dredged marine sediment. A significant improvement in shear strength and stiffness is achieved with the AAMSB-12MSS binder compared to AAMSB-12M, underscoring the role of silica availability in enhancing the mechanical properties of treated sediment.

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