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## **SUSTAINABLE REMEDIATION TECHNOLOGIES FOR CONTAMINATED MARINE SEDIMENTS: PRELIMINARY RESULTS OF AN EXPERIMENTAL INVESTIGATION**

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### **Abstract**

This paper deals with the relevant environmental issues concerning marine sediments contamination, for which there is the need for further development of sustainable remediation technologies. Among the in-situ remediation options, the reactive capping turns out to be an interesting approach. On the other hand, when the sediments must be dredged, the stabilization and solidification (S/S) treatments can represent an efficient and sustainable solution for the recovery of the materials, in the spirit of the circular economy. In the present paper, the first results of an on-going experimental investigation on remediation of the contaminated marine sediments from the Gulf of Taranto are presented. The research activities aim at assessing the effectiveness of both solutions as remediation options for marine sediments contaminated by heavy metals, polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs).

*Key words:* contaminated sediments, reactive capping, solidification/stabilization

*Received: March, 2018; Revised final: July, 2018; Accepted: September, 2018; Published in final edited form: October 2018*

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### **1. Introduction**

Aquatic sediments are identified as an ultimate receptor for many hazardous pollutants produced by the human activities (Zhang et al., 2016).

Sediment-bound pollutants pose major concerns for human health and the environment because these contaminants can re-enter the overlying water and become available to benthic organisms and subsequently pass into aquatic food chains. Thus, sediments act as both carriers and long-term secondary sources of contaminants for aquatic ecosystems.

Contaminated sediments remediation strategies may consist of in situ (i.e., treatments operating where the contamination is present with no sediments dredging) and ex situ options (i.e., treatments including sediments dredging or resuspension phenomena to some extent).

In situ remedial alternatives generally involve (i) Monitored Natural Recovery (MNR), which is essentially based on a deep knowledge of the system and its resilience capacity to reduce the risk over time through natural processes (De Gisi et al., 2017), (ii) in situ containment and treatment, in which either contaminated sediments are physically and chemically isolated from aquatic ecosystems or the contaminants in sediments are sequestered and degraded (Lofrano et al., 2017).

Ex situ remedial alternatives typically need several component technologies to dredging or excavation, transport, pre-treat, treat, and/or dispose sediments and treatment residues. The pollutants are extracted from the sediments or degraded through a series of chemical, physical, biological or thermal methods in specially designed reactors. Most ex situ remediation technologies developed for soil or waste

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can be used for dredged sediments (De Gisi and Notarnicola, 2016a).

Among the remediation options, In Situ Capping (ISC) can become, in some cases, one of the most sustainable options, i.e. in terms of efficacy, durability and economy. It consists in placing a layer of clean materials over the sediments to isolate the contaminant from the overlying water column and biota, to reduce contaminant flux into the biologically active part of the sediment, and to create new habitats for aquatic organisms (Reible, 2017). Conventional (passive) caps consist of a layer of clean neutral materials and they rely on containment, rather than treatment, of contaminated sediments. The cap may also include: geotextiles to enhance the layer separation or improve geotechnical stability, amendments (that are made up of chemically reactive materials, i.e. Active/Reactive Cap solutions) to boost the treatment efficacy, or additional layers to armour and keep the cap integrity or to help the development or restoration of the habitat characteristics. The use of chemically reactive materials allows the sequestration and/or degradation of the sediment contamination, reducing its mobility, toxicity, and bioavailability, thus performing both containment and treatment of the contaminated sediments. A variety of materials are proven to achieve the goals of cap; however, limited proved options exist for enhancing contaminant adsorption and degradation processes. Several laboratory experiments and recent field studies demonstrated that a layer of activated carbon, thick some centimetres, can effectively decrease contaminant flux from sediments to the overlying water (Abel and Akkanen, 2018; Gilmour et al., 2018; Hua et al., 2017).

Reactive capping is usually applied either for slightly polluted sediments or when there is the need to accelerate the lowering of contamination to get a faster risk mitigation. However, for heavily polluted sediments, or if dredging is compulsory to reach a desired bathymetric level, ex situ technologies for sediments remediation become the most appropriate choices. In these cases, the “beneficial reuse” of dredged materials is highly encouraged, e.g. Italian Ministerial Decree No. 173/2016 (MD, 2016). Therefore, treatments for decreasing pollutant concentration have become part of the sediments management process (Todaro et al., 2016).

Stabilization/solidification (S/S) of contaminated sediments seems to be an appealing technology for both the chemical and mechanical improvement (i.e. heavy metal immobilization, compressibility reduction and strength improvement). The S/S techniques can be different and vary depending on the target to be reached for the sediment reuse. They are based on adding chemical compounds to dredged material to achieve (i) the chemical immobilization of the contaminants, and (ii) the improvement of mechanically characteristics for reuse of the stabilize material as new construction material. S/S treatments generally do not remove the contaminants from the dredged material since the

pollutants are transformed into a less mobile and harmful species.

This study reports the first results of an experimental laboratory investigation, carried out on marine sediments contaminated by heavy metals, PAHs and PCB, to explore the sustainability of the following remediation technologies: in situ reactive capping and ex situ stabilization/solidification. In detail, the following issues have been investigated: (1) chemical characterization of contaminated marine sediments of Gulf of Taranto (South of Italy), (2) effect of Reactive Permeable Mats (RPM) with organoclay (OC) or activated carbon (AC) on the contaminant migration and (3) leaching of ex situ S/S treated sediments with several additives (i.e. Portland cement, lime, active carbon and organoclay).

## 2. Material and methods

### 2.1. Contaminated marine sediments

The sediments were sampled in the Gulf of Taranto, one of most polluted marine areas in Europe, declared Site of National Interest (SIN) (Italian Law, 1998) also because of serious contamination of marine sediments (Vitone et al., 2016).

The samples were taken up to depths of about 1.5 m from the seafloor, that is about the depth of interest in view of any mitigation solution. These were passed through a 2-cm sieve, homogenized by mixing and stored at 4 °C until use.

The standard protocols of ISPRA (the Italian Institute for the Environmental Protection and Research) was used for determining grain-size, moisture content and organic matter of sediments (ICRAM, 2001). The concentrations of heavy metals were obtained by ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) in accordance to the EPA method 200.8 (EPA, 1994). For the determination of the total PAHs and PCBs concentrations, as well as of each compound or homologue group, Gas Chromatography - Mass Spectrometry (GC-MS) EPA method 8275A (EPA, 1996) was used.

### 2.2. In situ option: laboratory experiments with reactive permeable mats

Reactive permeable mats with organoclay or active carbon were used. RPM represent an innovative remediation technique, consisting of a reactive layer (“mat”) containing one or more materials that are confined between two permeable geotextiles (Fig. 1a). The use of reactive mats for active ISC solutions can be useful to increase their adsorption capacity compared with that of the traditional caps (commonly made up of clean neutral materials: sand, silt, clay, and crushed rock debris), through the minimisation of cap thickness. Moreover, it has been verified that the same amendment is generally more effective when used in a cap than when placed directly into sediments (ITRC, 2014). The layers of synthetic geotextiles provide a

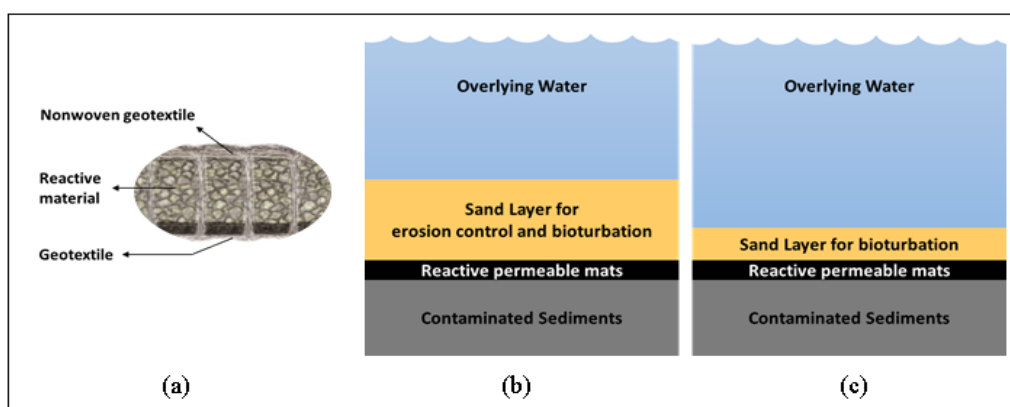
bioturbation barrier that prevents the mixing of amendments with the underlying sediments, allowing their uniform application, and reducing the effect of erosion processes (Olsta et al., 2006). However, their durability is conditioned by the site use; if anthropic activities are foreseen to be conducted in the site, a protecting layer of sand cannot be avoided (Fig. 1b). When possible, a new habitat layer can be directly overlapped on the mat (Fig. 1c).

A RPM 0.6 cm thick and a quantity of either Organoclay (OC) or Activated Carbon (AC) equal to 3 kg/m<sup>2</sup> have been used. OC is a modified clay that shows a specific adsorption behaviour towards PAH, free oil and grease (FOGs) and chlorinated hydrocarbons. It has been also verified that OC can have good potential for remediating different heavy metals under real environmental conditions (Meric et al., 2014). AC, which derives from thermal decomposition of various carbonaceous materials followed by an activation process, is probably the most widely used material among active substances (De Gisi et al., 2016b). Various laboratory studies showed that active sediment mixing with AC can

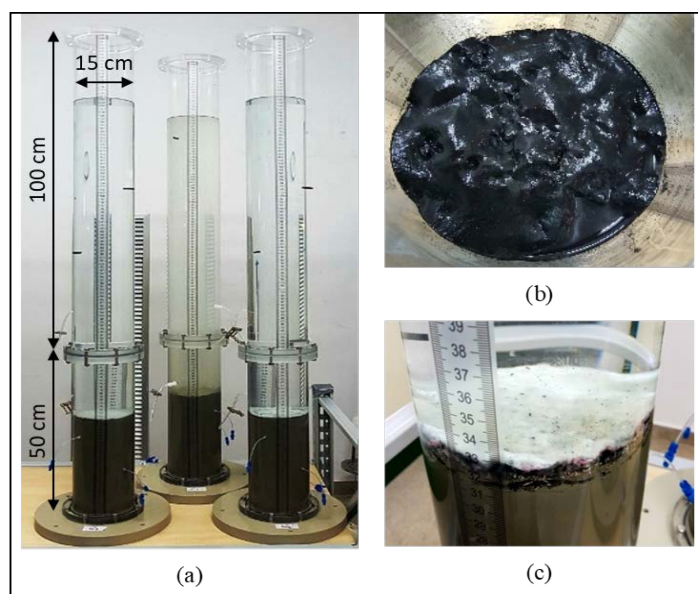
significantly reduce the aqueous concentrations, bioavailability, and bioaccumulation of PCBs, PAHs and several heavy metals (Choi, 2018; Gustafsson et al., 2017).

The experimental set-up (Fig. 2) was designed and assembled to test the efficacy of the reactive capping at lab-scale level. The set-up is intended to be an accurate physical model of the subaqueous site with contaminated sediment, reactive mat and water column in place. The columns used are 15 cm in diameter and 150 cm in length. They are made up of two sections of different length (50 cm and 100 cm) to facilitate handling and cleaning.

A porous plate at the base of the columns has been fixed to allow for water injections into the column through a 6.35 mm opening. Various sampling ports are present at different heights along the columns for chemical sampling of the effluent to be taken during the test. After every 20 days the water of each column has been sampled and analysed. The settlement of the top sediments has been recorded over time to follow the development of the consolidation process.



**Fig. 1.** (a) Reactive permeable mats (Zhang et al., 2016, modified), (b) active cap with sand protecting layer, (c) active cap with sand habitat layer



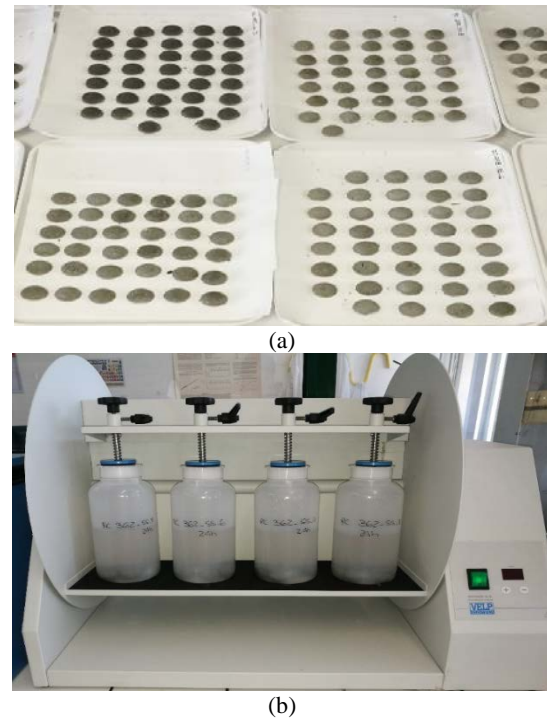
**Fig. 2.** Experimental set-up: (a) columns, (b) sediments, (c) reactive permeable mats

The mixtures were prepared by using different contents of several additives, namely CEM I 42.5 R Portland cement (C), lime (L), activated carbon (AC) and organoclay (OC). An 0.8 water/dry matter ratio (w) was used for all experiments; the value was based on the results of preliminary tests as well as the high clay content in sediment (Table 1). All the materials were initially mixed for 5 min with a standard mixer and, then, a steel trowel to ensure a homogeneous paste was used. In the casting phase, the prepared mixture was introduced into different plastic moulds with hemispherical shape. The samples, in the curing phase, were kept at  $20 \pm 5$  °C and 80% humidity (Fig. 3a). In Italy, as in some other EU countries, the reuse options of treated sediments are evaluated with leaching tests (UNI EN, 2004). To this aim, for each sample a 40-g portion was sampled and transferred to a polyethylene bottle.

Distiller water was added with a solid-liquid ratio of 1:10 by weight and the bottle was kept in rotation at 12 rpm for 24 hours using Rotax 6.8-Velp Scientifica (Fig. 3b).

To end of the 24 hours, a short retention time was given to the extraction vessels for the settlement of suspended coarse solids; then, the leachate was filtered for the removal of suspended solids. The soluble concentrations of heavy metals of interest (As,

Co, Cr, Ni, V and Zn) were analysed using ICP-OES.



**Fig. 3.** Samples (a) and leaching test (b)

**Table 1.** Mix design for recycling sediments by S/S treatment

| Mixture | Additive | Reagent          | A+R content | Water/dry matter |
|---------|----------|------------------|-------------|------------------|
| MIX_1   | Cement   | -                | 10%         | 0.8              |
| MIX_2   | Cement   | Activated Carbon | 15%         | 0.8              |
| MIX_3   | Cement   | Organoclay       | 15%         | 0.8              |
| MIX_4   | Lime     | -                | 10%         | 0.8              |
| MIX_5   | Lime     | Activated Carbon | 15%         | 0.8              |
| MIX_6   | Lime     | Organoclay       | 15%         | 0.8              |

**Table 2.** Physical-chemical properties of the sediments used for the tests

| Parameter              | Unit  | Value  | St. Dev. |
|------------------------|-------|--------|----------|
| pH                     | u. pH | 8.8    | 0.05     |
| Eh                     | mV    | -100.9 | 0.28     |
| Conductivity           | mS/cm | 4.1    | 0.95     |
| Moisture content       | %     | 44.8   | 0.23     |
| Ashes at 600°C         | %     | 87.3   | 2.50     |
| Organic matter content | %     | 12.3   | 2.50     |
| Sand fraction          | %     | 19.4   | -        |
| Silt fraction          | %     | 43.2   | -        |
| Clay fraction          | %     | 37.4   | -        |
| PAHs                   | µg/kg | 5389   | 300      |
| PCBs                   | µg/kg | 1669   | 100      |
| As                     | mg/kg | 12.3   | 0.4      |
| Co                     | mg/kg | 7.1    | 0.03     |
| Cr                     | mg/kg | 54.0   | 3.4      |
| Ni                     | mg/kg | 38.2   | 0.6      |
| Pb                     | mg/kg | 87.4   | 4.1      |
| V                      | mg/kg | 53.3   | 4.0      |
| Cu                     | mg/kg | 69.6   | 10.5     |
| Zn                     | mg/kg | 189.2  | 16.6     |

### 3. Results and discussion

The sediments used for the tests were characterised; particle size distribution and physical-chemical properties are reported in Table 2. According to the marine sediment management guideline in Taranto SNI (ICRAM, 2004), the sediments are to be considered contaminated because the contaminants concentrations exceeded the site-specific thresholds, even if below Italian national limit values (LD, 2006).

Column tests were carried out to assess the effectiveness of reactive capping materials in the sequestration of various heavy metals and organic contaminants in saltwater. Column water quality monitoring was performed for 20 days from the reactive permeable mat placement. The water samples were analysed for 8 heavy metals, PCBs and PAHs.

Heavy metals were detected in water samples (all close to 1  $\mu\text{g/l}$ ), but these values were statistically equal to those measured for pre-cap conditions. This shows that the metals are strongly bound on sediments and that the presence of reactive agents (i.e. AC and OC) not increased the rate of diffusion. The release of Cd, the most mobile metal and hard to stabilization with apatite, active carbon or other capping materials (Dixon and Knox, 2012; Kang et al., 2016), was effectively controlled by RPM.

PCB (29 congeners) concentrations in water samples were not detectable (MDL is approx. 0.001  $\mu\text{g/l}$ ). The hydraulic condition applied for the columns (no pore-water flow) found a minor short-term desorption of PCBs compared to laboratory tests conducted with continuously mixed reactors (Choi et al., 2014), suggesting that the contaminant transport depends on flow of porewater. Also, the low permeability of the fine-grained sediments used (Vitone et al., 2016) could have hindered the

desorption of contaminants. PAHs were detected in water inside the reactor, both for cap and no-cap conditions. Fig. 4 shows the PAH concentrations at 20 days after the start of test conditions. The results show that PAH concentrations in water decreased from initial 0.1  $\mu\text{g/l}$  to near 0.04–0.06  $\mu\text{g/l}$  for column apparatus with cap. It is also observed that PAHs in water increased to 0.17  $\mu\text{g/l}$  without the cap (Fig. 4). Therefore, after 20 days from the placing, RPM provided a reduction of the 60% (RPM with AC) and 40% (RPM with OC) of the original PAH contamination.

The results confirm that reactive amendments (such as organoclay and active carbon) sequester the organic contaminants and control their mobility to the water column (Knox et al., 2012). However, several studies also report concerns about the durability of the effectiveness of the active/reactive cap solutions (Lofrano et al., 2017; Zhang et al., 2016).

Regarding S/S treatment, the results of leaching tests showed that organic contaminants were not present in the eluates, while heavy metals were released with concentrations varying with the reagents percentages. Fig. 5 shows a comparison of the leaching of heavy metals for the several mix designs used for S/S treatments. The ratio between the 28 days heavy metals concentration ( $C_{28}$ ) and the law limit ( $C_{LAW}$ ) is reported for the six mixing solutions. Only for the samples named MIX\_2 (Cement + AC) and MIX\_3 (Cement + OC) all heavy metals concentrations are lower than the law limit (i.e.  $C_{28}/C_{LAW}$  is always lower than 1). The leached copper amount reached values more than the law limit (0.05 mg/l) in samples with lime (MIX\_4, MIX\_5 and MIX\_6). Moreover, in the samples without reagents (MIX\_1 and MIX\_4) the leached nickel amount reached values more than the law limit (0.01 mg/l).

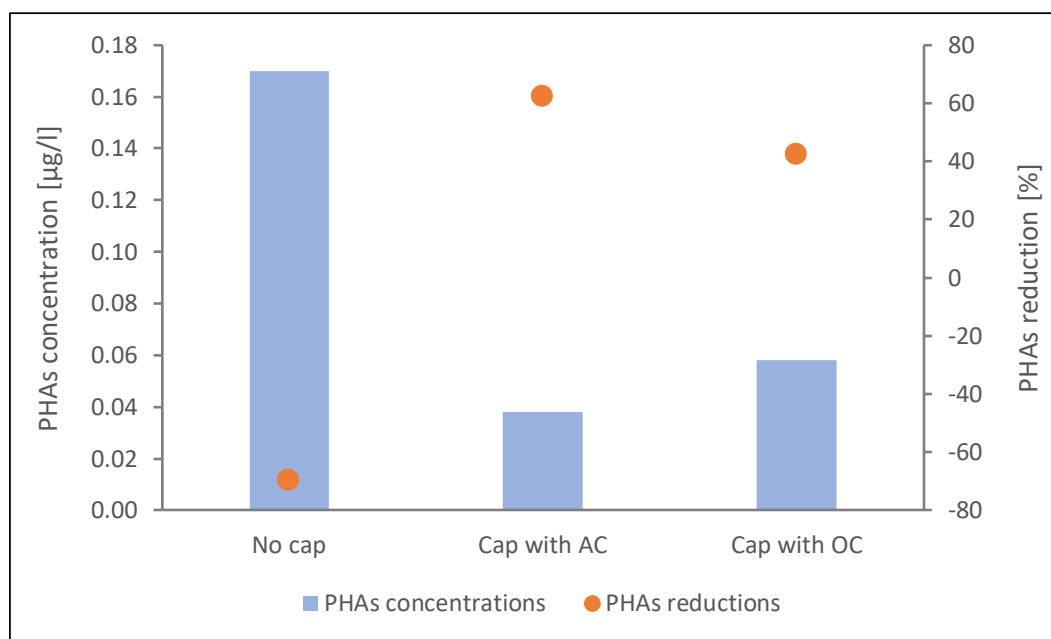


Fig. 4. PAHs concentrations in water after 20 days of capping test



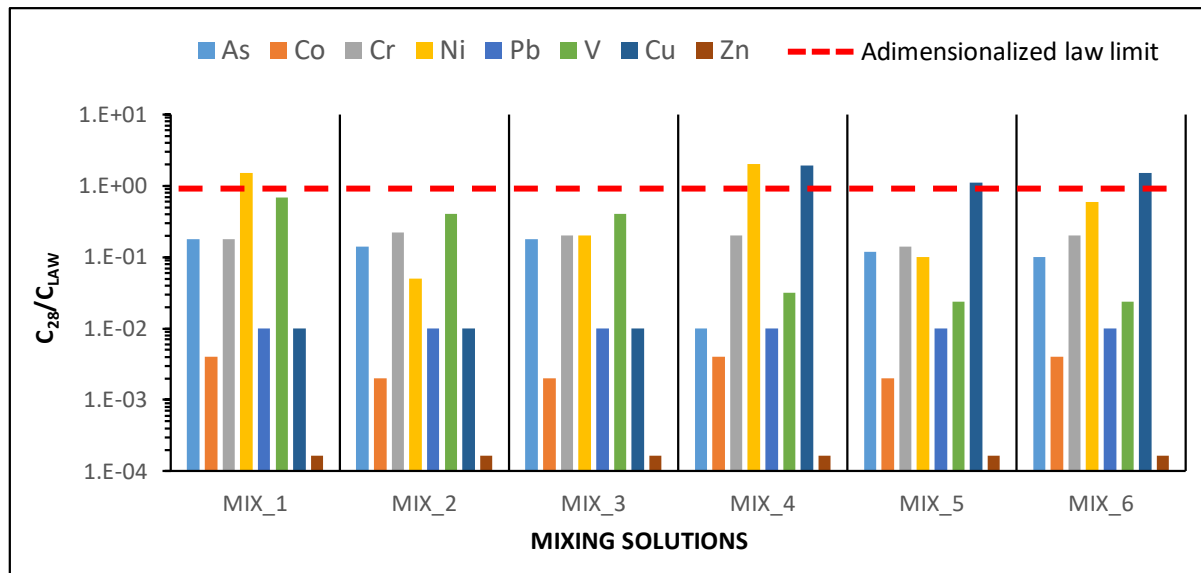


Fig. 5. Leaching at 28 days for each mix design and each heavy metal compared to law limit

The simplest form of treatment was obtained by the addition of either Portland cement or lime. The additives were used for the optimisation of performances, because the organic contaminants can interfere with the chemistry of binders, compromising the effectiveness of both the chemical and mechanical stabilization (Wang et al., 2013).

Therefore, for the mixtures with cement and OC or AC, the absence of leaching allows the beneficial reuse of contaminated sediments after S/S treatment.

The treated sediments can be recycled as aggregates for road construction (Pinto et al., 2011; Wang et al., 2012), cemented mortars (Couvidat et al., 2016), fill material and blocks (Tang et al., 2015; Wang et al., 2015) or raw material in brick production (Todaro et al., 2016; Cappuyns et al., 2015).

#### 4. Conclusions

The results of column tests have shown that the reactive capping could represent an interesting option in situ remediation technology for contaminated sediments. In particular, after 20 days from the placing of capping there is a reduction of the 60% (RPM with AC) and 40% (RPM with OC) of the original PAHs contamination; metals and PCBs are not released in water. However, the research needs to be further developed with special reference to the long-term effectiveness of the amendments.

This study also explored the possibility of re-using dredged contaminated sediments as resource via S/S treatment. After 28 days from preparation, only the sediments mixtures with cement and adsorbent materials (MIX\_2 and MIX\_3) have complied with the acceptance criteria for reuse in terms of leachability. In fact, the leached concentrations of copper, for the mixtures with lime, and the nickel, for the mixtures without adsorbents, have reached values more than the law limits. For the mixtures with

cement, the results show that the use of OC and AC has optimized the chemical stabilization.

However, to investigate the actual recovery potential of the S/S treated sediments, a mechanical testing program aimed at deepening the effectiveness of the treatments also in terms of sediment strength and compressibility needs to be carried out.

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