

Fig. 6. Fraction mobilised in water (left) and acetic acid (right) for the artificial waste-bearing bodies.

distribution is associated with a higher surface area, and because the leaching process is a surface reaction, it is likely that the higher mobilisation of HEs from the GR-AW body is related to its particle-size distribution.

Neither Sn nor Sb were mobilised in any of the test conditions, with the exception of two samples that exhibited a very low Sb release, both in water (fraction mobilised 0.002 for GPO-AW and 0.01 for NR-AW). Despite the high refractoriness of the oxides, both ions are insoluble over the entire pH range investigated, thus preventing the identification of differences in immobilisation mechanisms [107,108].

### 3.4. Ceramisation and element stabilisation

To outline general trends in the effectiveness of element stabilisation through the ceramisation process, this section also considers data related to the benchmark bodies (which also contains some amount of HEs, albeit at very low concentrations) and those containing BA (to assess possible effects of Cl and S, which occur at relatively high concentrations).

The mobilised fraction of HEs is plotted in Fig. 7 as a function of the amount of amorphous phase (and thus of the increasing temperature) for the unreacted body NR, the vitrified GR and the largely vitrified

GPO–GSTO. Two distinct behaviours can be identified: a group of HEs exhibiting a clear correlation between the fraction of the element mobilised and the amount of amorphous phase and another group with a less pronounced relationship. Co, Cr, Mo, Sr and V belong to the first group, exhibiting an inverse correlation between the two variables and similar ranges of fraction mobilised in basic and acidic solutions. The data indicate a direct relationship with the NBO/T of the vitreous phase in ceramic bodies: the lower the degree of polymerisation (higher NBO/T), the higher the fraction mobilised (Fig. S8, supplementary materials). This is consistent with the hypothesis that  $\text{Sr}^{2+}$  is more effectively retained as a charge compensator in highly polymerised glassy phases (and something similar may occur with  $\text{Co}^{2+}$ ). In the case of Cr, Mo and V, which are most likely present as oxyanions, it can be postulated that such complexes would be more easily released from less polymerised glasses. However, it should be noted that a correlation between leaching and NBO/T is not universally recognised [109].

The second group is composed of Ba, Cu, Pb and Zn, which exhibit the expected difference in the fraction of elements mobilised in basic (low or none) and acidic (high) environments. There is no clear trend as a function of the amorphous phase content. However, an inverse correlation with NBO/T can be observed (Fig. S9, supplementary materials): the fraction mobilised is greater when the degree of

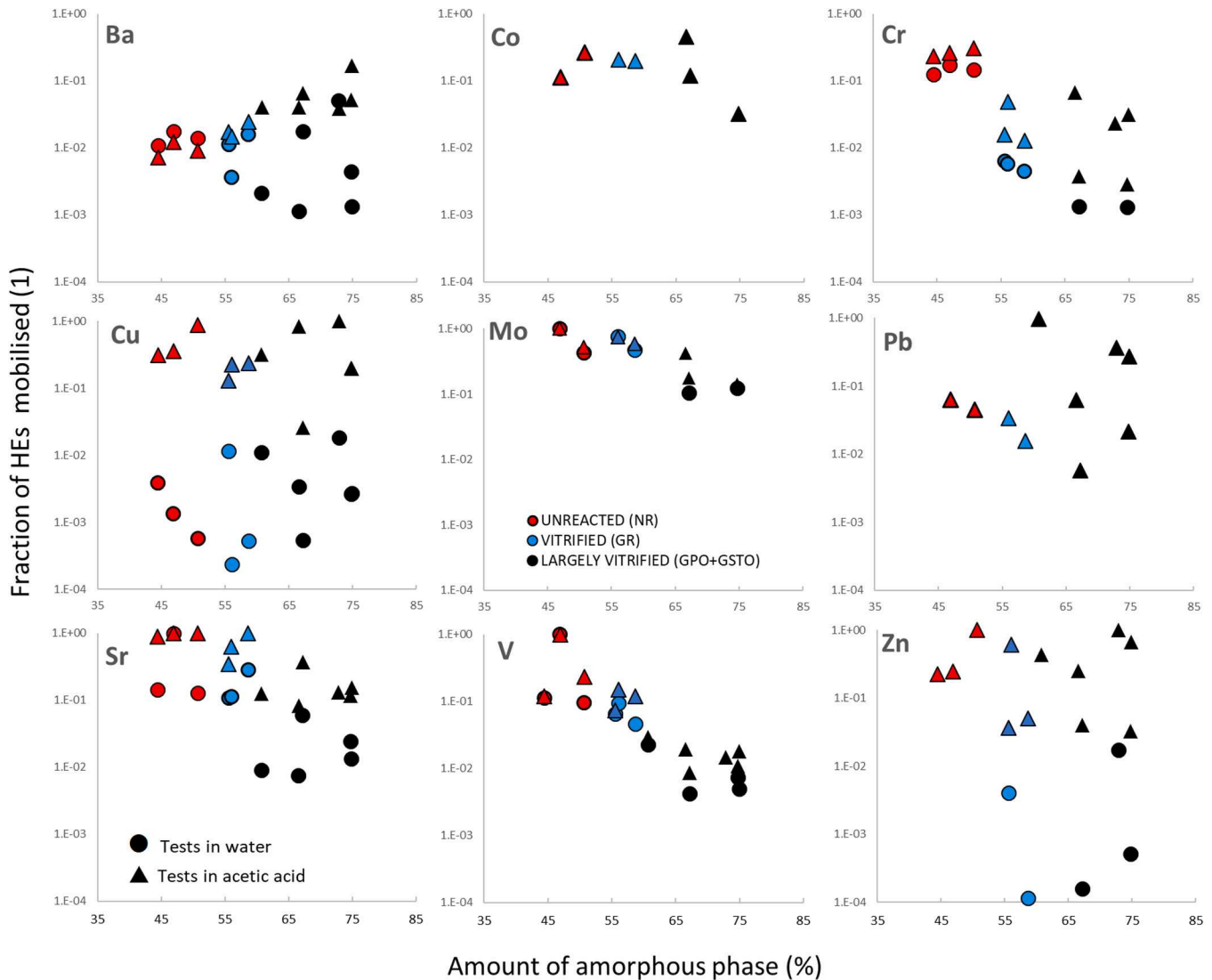


Fig. 7. Fraction mobilised in water (dots) and in acetic acid solution (triangles) of the benchmark bodies, artificial waste-bearing bodies and bottom ash-bearing bodies, plotted as a function of the amorphous phase content.

polymerisation is higher. This phenomenon can be attributed to the presence of  $Ba^{2+}$  (and similarly  $Pb^{2+}$ ) as GNM in porcelain stoneware. In addition, the lack of K-feldspar neo-formation precludes Ba (or Pb) from the most effective opportunity for incorporation into crystalline structures. The leaching behaviours of Zn and Cu appear to be somewhat correlated with the glass network modifiers. The higher is the fraction mobilised, the lower is the amount of GNM (Fig. S10, supplementary materials). This suggests that the latter may contribute to the stabilisation of  $Cu^{2+}$  and  $Zn^{2+}$  in low oxygen coordination number in aluminosilicate glasses [110]. Finally, Sb was not mobilised, except for two samples with a very low release ( $f_{Sb} = 0.002$  and  $0.01$  for GPO-AW and NR-AW, respectively). This corroborates the findings of previous studies, which reported  $f_{Sb} = 0.001\%$  at  $850\text{ }^{\circ}\text{C}$  [17] up to  $0.01\%$  at a temperature  $> 1300\text{ }^{\circ}\text{C}$  [111].

A comparison of the ion concentrations in water with the different types of waste indicated a systematically lower mobilised fraction from the bodies containing BA, whose higher concentrations of Cl and S have not induced a greater release of HEs than AW. However, the results of the leaching tests in acetic acid solution showed a higher mobilised fraction from the bodies containing BA, except for Ba and Sr. The differences observed in the behaviours of BA and AW were likely influenced by the presence of distinct HE-bearing phases, as previously reported in the literature [103-105].

The efficiency of immobilisation in water is plotted as a function of

the HEs concentration in ceramic batches in Fig. 8, where the results of this study were compared with the literature data relative to non-reactive, recrystallised and largely vitrified ceramic bodies. In general, silicate ceramics exhibit a high efficiency of HEs immobilization in water. This is particularly true for the elements Ba, Co, Cu, Pb, Sb, Sn and Zn.

*Barium* is typically stabilised in highly vitrified matrices, although there are instances of retention rates between 98% and 99% and only one sample below this value (GSTO  $\epsilon_{Ba} \sim 95\%$  at a low BaO concentration, 0.05 wt%). These efficiencies are only marginally inferior under acidic conditions. This corroborates some previously documented cases of relatively low immobilisation observed when BaO is  $> 0.1\%$  in the ceramic material [112]. These unsatisfactory cases may result from the absence of the most effective mechanisms for barium stabilisation. These mechanisms include the formation of feldspars during firing, which can accommodate  $Ba^{2+}$  in the crystalline structure [99], and the binding to the tetrahedral glass framework as a charge compensator when alkali and Ca are not sufficient to balance the available  $Al^{3+}$  [113].

*Chromium* is immobilised with high efficiency in largely vitrified bodies ( $\epsilon_{Cr} \geq 99.4\%$ ), confirming the results of the literature (Fig. 8). Red stoneware exhibits slightly lower performance than porcelain stoneware. Leaching in acid solution resulted in a slightly lower degree of immobilisation than in water. A warning signal was given by the scarcely reacted body (NR), regardless of the  $Cr_2O_3$  content, which had

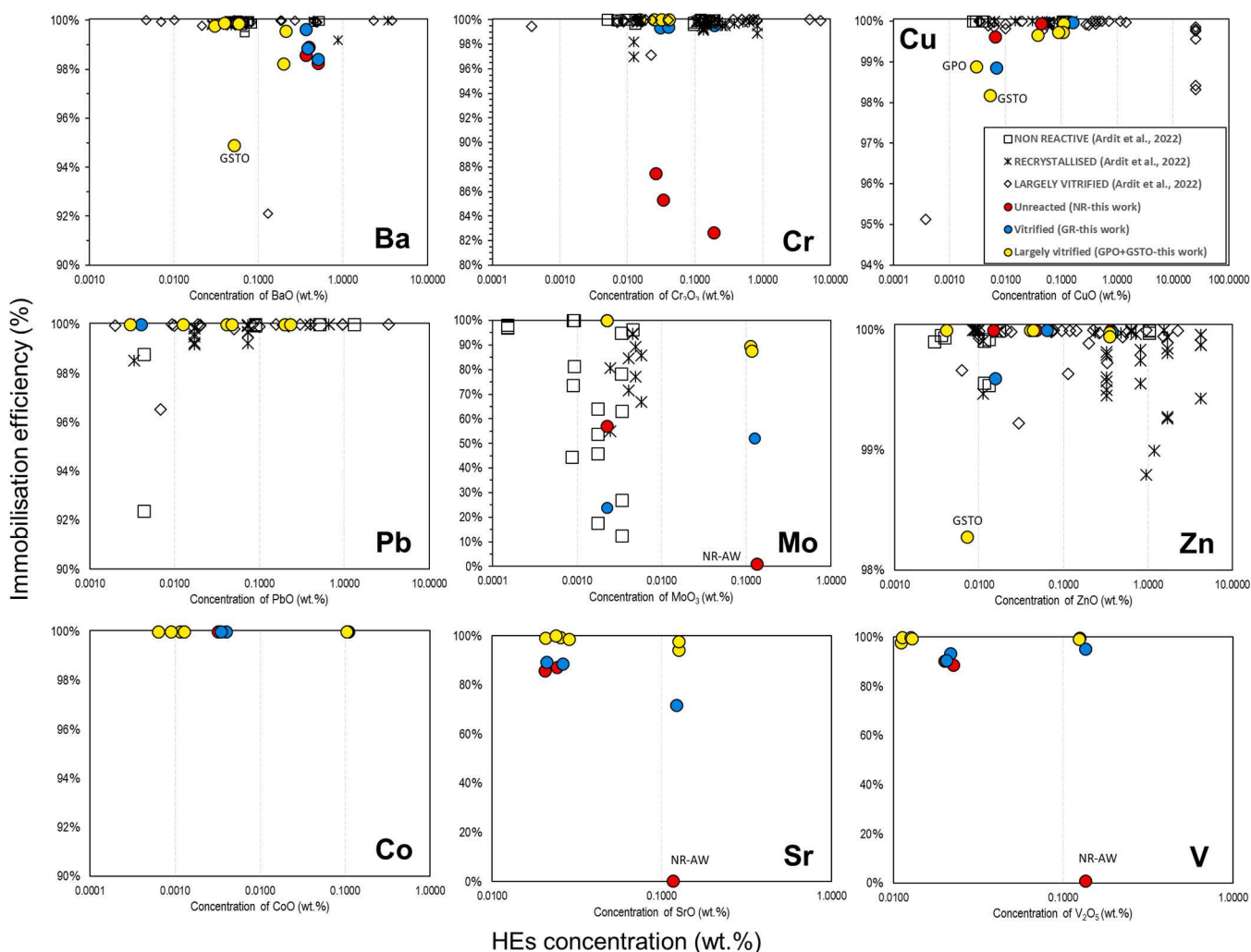


Fig. 8. Immobilisation efficiency in water plotted as a function of the HEs concentration (oxide wt%) in benchmark bodies, artificial waste-bearing bodies and bottom ash-bearing bodies. The results of this study are compared with the data collected by Ardit et al. [6] relative to non-reactive, recrystallised and largely vitrified ceramic bodies.

poor immobilisation efficiency ( $\epsilon_{Cr}$  82–88%). Thus, the amorphous phase present in the samples fired at 820 °C had a substantially lower Cr retention capacity than in aluminosilicate glasses formed in stoneware fired above 1100 °C (GR samples).

*Cobalt* exhibits complete immobilisation under basic conditions, regardless of the type of ceramic matrix. This is consistent with previously reported data indicating its high efficiency ( $\epsilon_{Co}$ ~99.7%) also for non-vitrified ceramics fired at 950 °C [12]. However, the acetic acid solution test indicated a slight loss of Co retention, the values of which range from 88% to 97% in porcelain stoneware and approximately 80% in red stoneware.

*Copper* is stabilised with high efficiency in vitrified bodies ( $\epsilon_{Cu}$  ≥ 98%), as previously demonstrated in other studies (Fig. 8). The degree of immobilisation is slightly more pronounced in red stoneware than in porcelain stoneware, which is likely associated with the presence of hosting phases (i.e. clinopyroxene and/or spinel). Low efficiency was observed for the CuO concentrations below 0.01% in the ceramic matrix and could be affected by the greatest experimental uncertainty. However, Cu is leached much more under acidic conditions, regardless of the degree of vitrification.

*Lead* is completely immobilised in vitrified ceramic matrices ( $\epsilon_{Pb}$ ~100%) at both low and high PbO concentrations, fully confirming what has already been observed in the literature. Some Pb was released from waste-free porcelain stoneware bodies under acidic conditions, i.e. at extremely low PbO concentrations.

*Molybdenum* is a critical case where there is a low- to -very -low immobilisation efficiency under both basic and acidic conditions. Furthermore, the degree of Mo stabilisation decreases with the degree of vitrification. Porcelain stoneware bodies with AW exhibit  $\epsilon_{Mo}$ ~88%, whereas vitrified GR-AW  $\epsilon_{Mo}$ ~52% and NR-AW exhibit  $\epsilon_{Mo}$ ~1%. These data confirm the state of the art picture (Fig. 8) and extend it to MoO<sub>3</sub> concentrations at approximately 0.1%. The difficulties in Mo stabilisation may be attributed to the formation during firing (below the detection limit of XRD) of alkaline and alkaline earth molybdates with high solubility in water [6,114].

*Strontium* is efficiently immobilised in porcelain stoneware, particularly for SrO concentrations below 0.1% in the ceramic bodies. It appears that favourable for Sr retention are the formation of plagioclase during firing and the ability to host Sr<sup>2+</sup> in glass as a charge compensator for the Al<sup>3+</sup>-Si<sup>4+</sup> substitution among glass network formers. However, the Sr retention was considerably lower for the body made of red clay and when leaching was carried out under acidic conditions. These observations appeared to be somewhat at odds with the findings of previous studies, which indicated a high Sr immobilisation ( $\epsilon_{Sr}$  > 99.3%) regardless of the pH and SrO concentrations [37,38,115].

*Vanadium* immobilization efficiency on the ceramic matrix exhibits a clear differentiation. It is notably high for porcelain stoneware ( $\epsilon_V$  > 99%) but considerably lower for red clay bodies, particularly those fired at low temperatures. This pattern is observed across a range of pH values in leaching solutions. This corroborates the findings of previous studies on V leaching from ceramics, which have documented a high degree of immobilisation under acidic conditions [38,116] but a comparatively lower performance in water [117].

*Zinc* is retained in vitrified ceramic bodies with high efficiency ( $\epsilon_{Zn}$  close to 100%), with the exception of a single case ( $\epsilon_{Zn}$ ~98%) associated with a very low concentration in the body (ZnO < 0.01%). However, Zn<sup>2+</sup> can be released under acidic conditions. The main stabilisation mechanism in vitrified bodies is probably the incorporation within the glassy phase, where Zn<sup>2+</sup> ions are tetrahedrally bonded to oxygens with short bond distances.

The hazard quotient ( $HQ_{HE}$ ) is plotted in Fig. 9 for different HEs and types of silicate ceramics. In most of the cases analysed, the calculated values are relatively low, indicating that the concentrations of the released HEs are within the limits set by regulatory authorities for inert materials. This is particularly true for Cu, Zn and Pb, as well as Ba, although with a slightly higher hazard quotient (0.18 on average). A

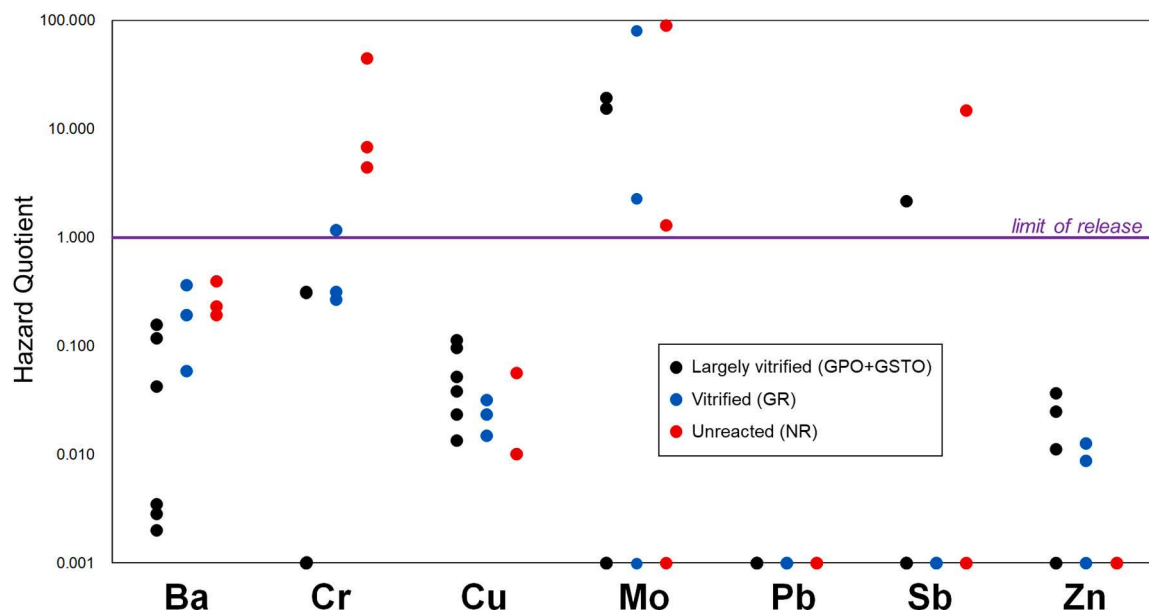
different situation was observed for Sb, Cr and particularly Mo, which show values exceeding the threshold set by the regulation, especially for NR-AW. This picture is not dissimilar to that sketched in the literature, indicating that Mo is the most challenging case [6].

#### 4. Conclusion

This study aimed to evaluate the practicality of employing ceramisation for inertising HEs potentially originating from secondary raw materials, which are increasingly used in the industrial ceramic sector. To this end, an artificial waste (AW) and a real bottom ash (BA) containing different HEs were incorporated into distinct types of silicate ceramics: vitrified, highly vitrified and largely unreacted. Quantitative mineralogical analyses of the fired ceramic bodies confirmed the formation of crystalline/amorphous phases capable of hosting different HEs, with the majority occurring in vitrified and largely vitrified bodies. Contrarily, the unreacted one, fired at a low temperature (820 °C), exhibited the prevalence of residual phases. The microstructural study provided insights into the reactivity of the two waste materials during the firing process. The AW effectively interacted with the ceramic matrices, leading to the incorporation of HEs in the neof ormation phases. Conversely, the interaction between the BA and the ceramic bodies appeared to be relatively low. The persistence of unreacted or minimally reacted BA particles was mainly attributed to the refractoriness of its constituents. Furthermore, the strong chemical and mineralogical heterogeneities of this waste (where the same element can be present in many different forms) made the interpretation of its behaviour challenging. For these reasons, the leaching data on BA-based ceramic bodies reflect the nature of the waste rather than its interaction with the ceramic matrices. Nevertheless, some general trends can be identified from the obtained results.

- Vitrified ceramic bodies exhibited high immobilisation efficiency for Ba, Co, Cr, Cu, Pb, Sb, Sn and Zn. This was also observed for Sr and V but limited to highly vitrified bodies.
- Mo is the only HE considered here that exhibited poor retention in ceramic matrices.
- A greater release of Co, Cu, Pb and Zn was observed in acidic environments, as compared with a neutral or alkaline environment, in the leaching tests conducted in water. This was mainly due to the predominance of water-soluble forms at the pH values of the acetic acid test.
- The degree of immobilisation of Co, Cr, Mo, Sr and V was enhanced by increasing the degree of vitrification and the firing temperature of the ceramic bodies. This appeared to be associated with the resulting greater polymerisation of the vitreous phase for several reasons. First, Sr<sup>2+</sup> can act as a charge compensator and thus contribute to the insertion of Al<sup>3+</sup> into the tetrahedral network, making it somewhat more difficult to leach out. Second, it can be postulated that Cr, Mo and V, which are presumed to exist in oxyanion form, are more readily leached from samples fired at lower temperatures owing to their apparent reduced binding to the glass network formers.
- In the case of Ba, Cu, Pb and Zn, it was observed that immobilisation was less efficient (mostly in an acidic environment) as the degree of polymerisation of the glassy phase increased. The reason for the behaviour of Ba (probably also Cu and Pb) could be that it is not bound to the tetrahedral network and is relegated among the glass network modifiers. This is due to the fact that alkali and other alkaline earth elements are more selectively involved as charge compensators in aluminosilicate vitreous phases. Zinc may be more loosely retained because although in tetrahedral coordination with oxygen, it is not part of the glass network formers.
- The mobilised fraction of Ba, Cu, Pb and Zn in water (according to EN 12457-2:2002) was consistently well below the limit required for inert waste. Cr, Sb and Mo exceeded the limit. The cases for Cr were represented by the unreacted bodies fired at low temperature, which





**Fig. 9.** Hazard quotient in benchmark bodies, artificial waste-bearing bodies and bottom ash-bearing bodies. The violet line represents  $HQ = 1$ ; all points on the top of this line correspond to HE concentrations in the eluates that exceeded the limits established by Regulation 2003/33/EC for inert materials.

showed much lower immobilisation efficiency than the vitrified and highly vitrified matrices (85.2% vs. 99.8%, respectively). This could be related to the oxidation of  $Cr^{3+}$  leading to the formation of  $[Cr^{6+}O_4]^{2-}$  complexes, which are easier to mobilise. For Sb, although characterised by high immobilisation efficiency (~99%), two samples exceeded the legal limits. As aforementioned, Mo was the main element of concern. It has been suggested that these criticalities were likely to be associated with the formation of oxyanionic complexes, including  $[Mo^{6+}O_4]^{2-}$ ,  $[Mo^{5+}O_4]^{3-}$  and  $[Sb^{5+}O_4]^{3-}$ . These complexes are challenging to immobilise in silicate matrices.

This study yielded successful results in the immobilisation of HEs through the ceramisation process, with the majority of these results observed in vitrified (red stoneware) and highly vitrified (porcelain stoneware) ceramic products. In particular, the immobilisation efficiency of porcelain stoneware is higher than that of red stoneware, whereas the differences observed between the porcelain stoneware matrices appeared to reflect the variation in the amount and degree of polymerisation of the vitreous phase. The potential impact of this study includes a paradigm shift from the empirical approach commonly employed in waste recycling to a new batch design strategy. Similar to all academic research, this study provides the basic knowledge needed before moving on to higher TRL. We believe that this work, and others made in this perspective, will help address eco-design towards safer and knowledge-based practices.

### Environmental implication

The study of the ceramisation process is crucial for assessing the immobilisation of HEs, particularly in the ceramic industry, which is increasingly adopting recycling practices according to circular economy principles. The work provides a quantitative analysis of the process, evaluating the effectiveness of incorporating secondary raw materials (containing hazardous constituents) into vitrified and highly vitrified silicate ceramic bodies. Valuable insights are provided on how to mitigate environmental risks associated with the introduction of potentially harmful elements. This comprehension is essential for improving waste recycling in the ceramic industry and promoting sustainable practices in accordance with the goals of circular economy.

### CRediT authorship contribution statement

**Mario Tribaudino:** Writing – review & editing, Resources, Funding acquisition. **Giuseppe Cruciani:** Writing – review & editing, Funding acquisition, Conceptualization. **Michele Dondi:** Writing – review & editing, Writing – original draft, Funding acquisition, Conceptualization. **Chiara Zanelli:** Writing – review & editing, Supervision, Investigation, Conceptualization. **Chiara Molinari:** Writing – review & editing, Writing – original draft, Methodology, Investigation. **Matteo Ardit:** Writing – review & editing, Writing – original draft, Investigation, Conceptualization. **Sonia Conte:** Writing – review & editing, Writing – original draft, Visualization, Investigation, Data curation, Conceptualization. **Luciana Mantovani:** Writing – review & editing, Writing – original draft, Visualization, Investigation.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data availability

Data will be made available on request.

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### Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jhazmat.2024.134657](https://doi.org/10.1016/j.jhazmat.2024.134657).

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