LEACHING BEHAVIOUR OF SINTERED CONTAMINATED MARINE SEDIMENTS

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ABSTRACT

Contaminated marine sediments from Cantabrian estuaries have been compacted and fired to produce sintered ceramic materials. The effect of sintering on the leaching behaviour has been investigated by means of the comparison of concentrations of species proposed by the European Waste Landfill Directive present in leachates and regulatory limits for them. The impact of the specimens on the environment is assessed.

In order to reach this objective, unfired and fired samples of marine sediments and clay were subjected to the Compliance Leaching Standard Tests EN 12457 1-2 using different liquid to solid ratios (L/S = 2 and 10). The concentrations of Se, As, Cr, Sb, Zn, Cd, Pb, Ni, Ba, Mo, Cu, fluoride, chloride and sulphate in leachates were analyzed, and the results compared with regulatory limits collected in 2003/33/CE Directive. The results revealed that the firing process decreases the leaching of the species analyzed, except for As. The comparison of the metal and metalloid concentrations in leachates and regulatory limits showed that all species fulfilled them, excepting As, fluoride, chloride and sulphate in unfired specimens. Thus, the potential environmental risk in relation to the leaching behaviour associated to the sintered contaminated marine sediments can be considered to be low versus to commercial clay.

Keywords: sediment, clay, leaching behaviour, sintered ceramics, environmental risk

INTRODUCTION

Coastal sediments act as temporary or long-term sinks for many classes of anthropogenic contaminants and, consequently, are the source of these substances in the ocean and biota [1]. According to the European Waste Catalogue, dredged harbor sediments are classified as waste material [2]. Thus, marine disposal has to be avoided and sediments must be dewatered in drainage ponds, and then deposited in landfills, which is an unsatisfactory solution from both the economic and environmental points of view [3].

The incorporation of waste materials into production cycles is an interesting alternative in modern industry, which leads to economic profit and considerable reduction in environmental impact. The incorporation of waste materials into brick manufacturing has become more and more consolidated [4-12] Marine sediments can be used as a substitute for clay in ceramic processes to obtain building materials [13,14]. During the sintering process, organic contaminants are destroyed and remaining metal contaminants are either volatilized or converted into stable immobile compounds. However, homogeneity and sufficient availability are preconditions for using marine sediments as a raw material in any technological process.

Previous studies have shown that sediments from the Santander Bay, Northern Spain, contain significant concentrations of heavy metals and organic pollutants [15-18]. Thus, the pollution of these sediments represents a high risk, making their treatment and confined disposal necessary in order to manage them properly.

Previous works have shown that it is possible to obtain dense sintered compacts from highly contaminated marine sediments dredged from Cantabrian estuaries [3]. Composition, thermal and mechanical behaviour of sintered ceramic bodies from contaminated sediments were evaluated. It was concluded that the studied marine sediments can be used as clay replacement in brick production.

The feasibility of the manufacturing process using wastes as raw material in the ceramic industry will be carried out not only in relation to technological and thermal properties, but also taking into account environmental behaviour.

Therefore, the leaching behaviour of these new ceramic bodies produced gives information about different stages of the life cycle of the brick. In this paper, the environmental risk of clay and sediment samples, before and after of their thermal treatment, has been assessed. In order to reach this aim, unfired (called granular) and fired (called sintered) samples of clay and contaminated marine sediment are submitted to the Compliance Leaching Standard Tests UNE-EN 12457 1-2 [19, 20], proposed in the European Directive on the Landfill of Waste, 2003/33/CE. Leaching tests used two liquid/solid ratios, L/S=2 and 10, which simulate closed and open landfill conditions, respectively. In this case, leaching data provided information about the end life stage of ceramic bodies, when a building is pulled down and the bricks become a demolition waste.

Leachates obtained in these tests were analyzed both to evaluate the influence of L/S ratio and sintering process over the leaching behaviour and to compare the chromium, zinc, cadmium, nickel, barium, molybdenum, copper, lead, arsenic, antimony, selenium, fluoride, chloride and sulphate concentrations against the threshold limitations stated in the European regulation.

MATERIALS AND METHODS

Raw materials and specimens manufacturing

The Santander Bay is a coastal area located in the north of Spain (Cantabrian Sea), which represents one of the most dynamic natural environments in the Cantabrian Region. Used in this work were two marine sediments from Raos and Astillero, areas of the Santander Bay. These sediments were collected, homogenized and dried at room temperature.

The resulting powders, with a humidity of 4%, were compacted at 100 bar to form disc-shaped specimens (4 cm in diameter and 0.1 cm in height approximately). The compacted specimens were sintered in a muffle furnace at their optimum sintering temperatures, obtained in previous work [3], using a ramp rate of 4 ºC/min up to 1125 ºC for Astillero and clay samples, and 1150 ºC for Raos samples, and these temperatures were held for 1 h. The total element content of fired specimens was determined in Actlabs Ltd. (Ontario, Canada), with the purpose of characterizing them prior to their leaching behaviour evaluation. Total element content was extracted by means of an acid digestion with a mixture of hydrochloric, nitric, perchloric and hydrofluoric acids. A microwave was employed to attend the extraction. After that, the total content was determined by using FUS-ICP and AR-MS, depending on the elements.

2.2. Leaching tests

Equilibrium leaching test UNE-EN 12457 is a European Standard batch leaching test for leaching of granular waste materials and sludges at Compliance Level. In this work, two procedures have been performed for material with particle size 1-4 mm, at a liquid/solid ratio of 2 l/kg, “EN12457-1” or 10 l/kg, “EN 12457-2”. In the two tests, the leachant is demineralized water and the contact time be tween the leachant-solid material is 24 h. The unfired and fired clay and sediments, called granular and sintered samples respectively, are subjected to the leaching tests.

The leachates were analyzed to determine the concentrations of metals and anions considered in EU Waste Landfill Directive 2003/33/CE, in order to compare them with regulatory limits.

Metal concentrations of chromium, zinc, cadmium, nickel, barium, molybdenum and copper were analyzed by using Inductively Coupled Plasma-Atomic Emission Spectrometry (Perkin Elmer Plasma 400). The lead concentration was analyzed by Atomic Absorption Spectrometry in a Graphite Chamber (Perkin Elmer HGA 700). Metalloid concentrations of arsenic, antimony and selenium were analyzed by Hydride Generation. Amounts of fluoride, chloride and sulphate were determined by using Ionic Chromatography (Dionex DX-120 Ion Chromatograph).

RESULTS AND DISCUSSION

3.1. Raw materials characterisation

Sediments and clay samples used in this study were characterized according to Action Levels (ALs) proposed by CEDEX [21]. ALs represent chemical-specific concentration values that define the quality of the dredged material being assessed. These approaches establish different management options depending on the quality status of the sediment under consideration [22]. The approach involving 2 ALs can be summarized as follows: concentrations of contaminants in the material below the lower action level AL1 mean that disposal at sea would generally be permitted; those falling between AL1 and AL2 represent moderate contamination, and the dredged material would require further investigation before disposal could be permitted; those concentrations above AL2 indicate that the dredged material is highly contaminated and disposal at sea would generally not be permitted. In Table 1 total element content and ALs are compared in order to establish the category in which the raw material can be included.

TABLE 1 - Action Level (AL1 and AL2) values, total element content of unfired sediments and quality status of them referred to the species under consideration

	Pb				Cu			
	AL 1	AL 2	(mg/L)	Quality status	AL 1	AL 2	(mg/L)	Quality status
Clay Raos	120	600	18.8	Low	100	400	21.9	Low
			266	Medium			93	Low
Astillero			125	Medium			23.1	Low
	Zn				Cr			
	AL 1	AL 2	(mg/L)	Quality status	AL 1	AL 2	(mg/L)	Quality status
Clay			82.6	Low			21.9	Low
Raos	500	3000	630	Medium	200	1000	59.6	Low
Astillero			430	Low			48.1	Low
	As				Ni			
	AL 1	AL 2	(mg/L)	Quality status	AL 1	AL 2	(mg/L)	Quality status
Clay			18.8	Low			25.1	Low
Raos	80	200	22.6	Low	100	400	25.6	Low
Astillero			68.2	Low			31.5	Low
	Cd							
	AL 1	AL 2	(mg/L)	Quality status				
Clay			0.2	Low				
Raos	1	5	1	Medium				
Astillero			1.5	Medium				

The sediments from Raos have amounts of Cd, Cu, Cr, As and Ni below the AL1, while amounts of Pb and Zn are between AL1 and AL2. Sediments from Astillero have amounts of Cu, Zn, Cr, As, Ni and Cd below AL1, while amounts of Pb are above AL1, but below AL2. Although the content of some metals is below AL2, it is actually upper AL1, and so the sediments can be considered as category II ones, which implies materials with medium contamination. Thus, this dredged material requires a more exhaustive study prior to its controlled disposal into the sea. In contrast, the metal content of clay is below AL1 in all the species analyzed, so this material can belong to category I, less restrictive than category II.

Leaching behaviour

Leachates from UNE-EN 12457 test are analyzed in order to determine the concentrations of Cr, Zn, Cd, Ni, Ba, Mo, Cu, Pb, As, Sb and Se, considered in the Waste Landfill Directive. As, Se and Sb will be treated more carefully, due to their relatively high concentrations.

The influence of L/S ratio and firing process over leaching has been analyzed. Furthermore, concentrations and regulatory limits collected in 2003/33/CE have been compared.

The firing process has a specific relevance in leaching, varying the quantity of species in leachates. This variation is due not only to the gaseous release during firing process, but rather due to the fact that the sintering process can immobilize species in the ceramic matrix and decrease their leaching. Thus, only the influence of the firing process over the immobilization of species, without gaseous release consideration, is carried out.

Metal concentrations of Cr, Zn, Cd, Ni, Ba, Mo, Cu and Pb, as well as the pH values obtained in leaching tests, are shown in Table 2.

In all elements analyzed, the larger the liquid to solid ratio, the less the concentration of metal leached. Besides, leaching in granular samples is greater than in sintered samples. Thus, the sintering process decreases the leaching of these species, except that of Ba and Mo. These parameters have become slightly more mobile after thermal treatment only in case of Raos sediments.

The comparison between concentrations in leachates and required limits shows that granular samples from Raos overcome the regulatory limits for Zn, Ni and Pb in leaching tests with L/S=2, while sintered specimens of this location exceed the limit for Mo in this liquid to solid ratio. Granular samples from Astillero are above the limits for L/S=2 in Ni, Mo and Pb. Granular clay samples generate metal concentrations exceeding the required limits only for Mo in L/S=2 and 10 and for Zn in L/S=10.

Figure 1 shows the results of the analysis of the leachates obtained in the UNE-EN 12457 equilibrium leaching test performed both in granular and sintered specimens for As, Sb and Se.

If the influence of liquid to solid ratio over leaching is studied in granular samples, it can be seen that the higher the liquid to solid ratio, the lower the concentration of As, Se and Sb leached, except for As in clay and Se in Raos, since the values are very similar. In sintered samples, an increase in the liquid to solid ratio implies a decrease in the concentration of As and Sb leached, but an increase in concentration of Se, except for the Astillero sample.

Table 2. Metal concentrations (mg/l) and pH values in leachates

		L/S=2			L/S=10		
		Clay	Raos	Astillero	Clay	Raos	Astillero
Cr	G	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
	S	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
	L	0,1			0,05		
Zn	G	0,62	2,29	0,94	0,45	< 0,3	< 0,3
	S	< 0,3	< 0,3	< 0,3	< 0,3	< 0,3	< 0,3
	L	1			0,4		
Cd	G	< 0,004	0,011	0,009	< 0,004	< 0,004	< 0,004
	S	< 0,004	< 0,004	< 0,004	< 0,004	< 0,004	< 0,004
	L	0,015			0,004		
Ni	G	< 0,04	0,12	0,11	< 0,04	< 0,04	< 0,04
	S	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04
	L	0,1			0,04		
Ba	G	0,09	< 0,07	< 0,07	0,07	< 0,07	< 0,07
	S	< 0,07	0,45	0,07	0,07	0,19	< 0,07
	L	3,5			2		
Mo	G	0,31	< 0,027	< 0,027	0,27	< 0,027	< 0,027
	S	< 0,027	0,63	1,02	< 0,027	< 0,027	< 0,027
	L	0,15			0,05		
Cu	G	< 0,035	0,31	0,05	< 0,035	0,09	< 0,035
	S	< 0,035	< 0,035	< 0,035	< 0,035	< 0,035	< 0,035
	L	0,45			0,2		
Pb	G	< 0,05	0,13	0,12	< 0,05	0,62	< 0,05
	S	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
	L	0,1			0,05		
pH	G	7,62	7,00	7,42	7,21	7,71	7,74
	S	10,50	10,05	10,39	10,39	9,80	10,17

G: granular samples; S: sintered samples; L: Waste Landfill Limit

As can be seen in Figure 1, the sintering process increases the leaching of As in all locations. This fact is related to the solubilization of anionic species, such as As, which are highly dependent on the pH [2, 23]. Leaching of anionic species is favoured in extreme conditions, at acidic and alkaline pH values. The pH values obtained in the leaching tests carried out in fired samples are higher than those of granular samples, due to the decomposition of carbonates during the firing process, generating calcium hydroxides that increase pH values. Arsenic (V) is the dominant dissolved arsenic-species in natural waters, and has a high adsorption affinity to oxides and hydroxides of Fe and Al. It has been observed that the strong leaching of Al and Fe at pH 11 led to mobilization of formerly adsorbed arsenic [24]. Additionally, the As content of the harbor sediments can be in the same order of magnitude than clays [2]. Therefore, clays and sediments could cause similar leached concentrations of As.

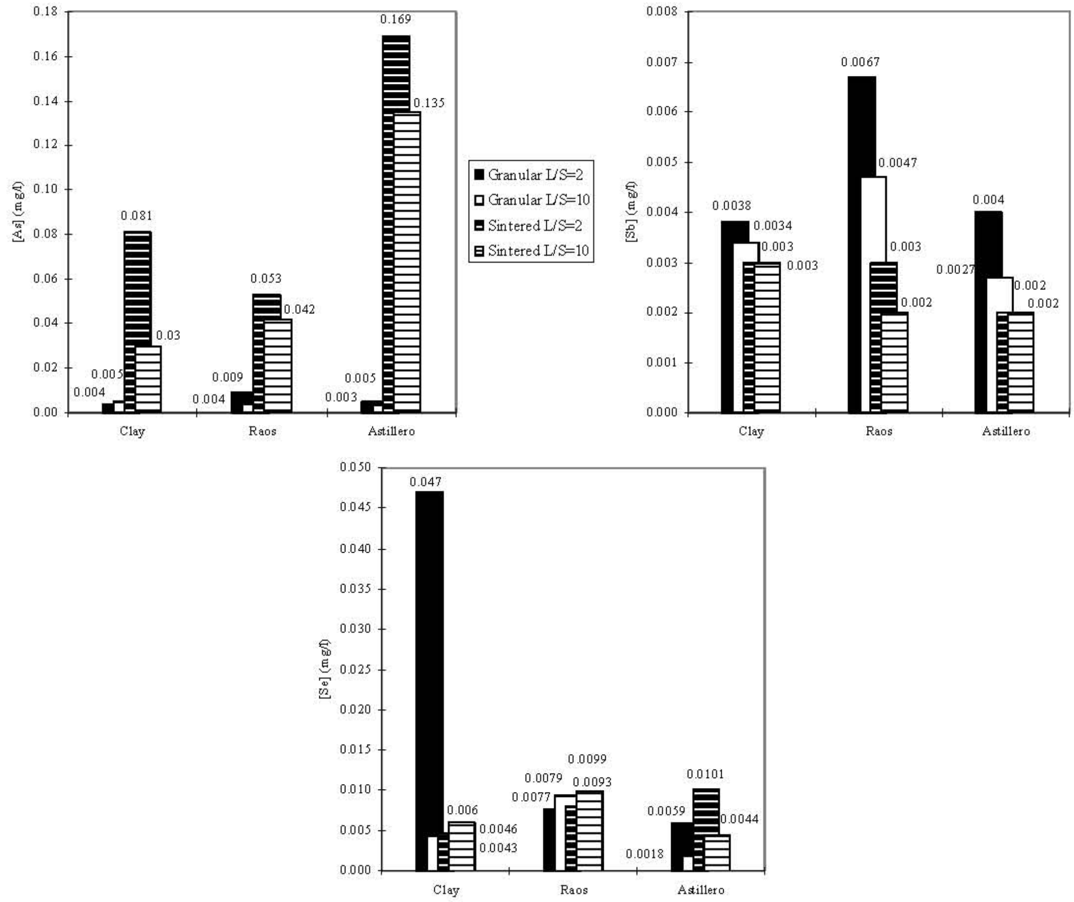

FIGURE 1 - Concentrations of As, Se and Sb in leachates obtained in UNE-EN 12457 test for clay, Astillero and Raos samples.

In contrast, the sintering process decreased Sb leaching for all specimens analyzed. The behaviour of Se is dependent on both the origin of the sample and the liquid to solid ratio employed. Leaching tests carried out with L/S =2 generate leachates with greater concentrations of Se leached, related to granular samples from the Raos and Astillero ones. Leaching tests developed with L/S=10 generate higher amounts of Se leached in clay, Astillero and Raos.

In order to establish the potential risk of both unfired and fired sediments and clay, leaching values and regulatory limits, stated in 2003/33/CE, are compared. Figure 2 shows a dimensionless concentration for granular and sintered samples, as well as the required limits. Leaching tests carried out in granular samples reveal that in most cases leaching rates of As, Sb and Se are below the required limits both for L/S ratios 2 and 10, excepting Se in clay. Sintered samples meet required limits for Sb and Se. Even so, sintered samples present concentrations of As leached which exceed the limits for L/S=2 in all samples, and for L/S=10 in sediments from Astillero. Therefore, it can be concluded that the firing process reduces the leaching of Se and Sb, and increases the leaching of As.

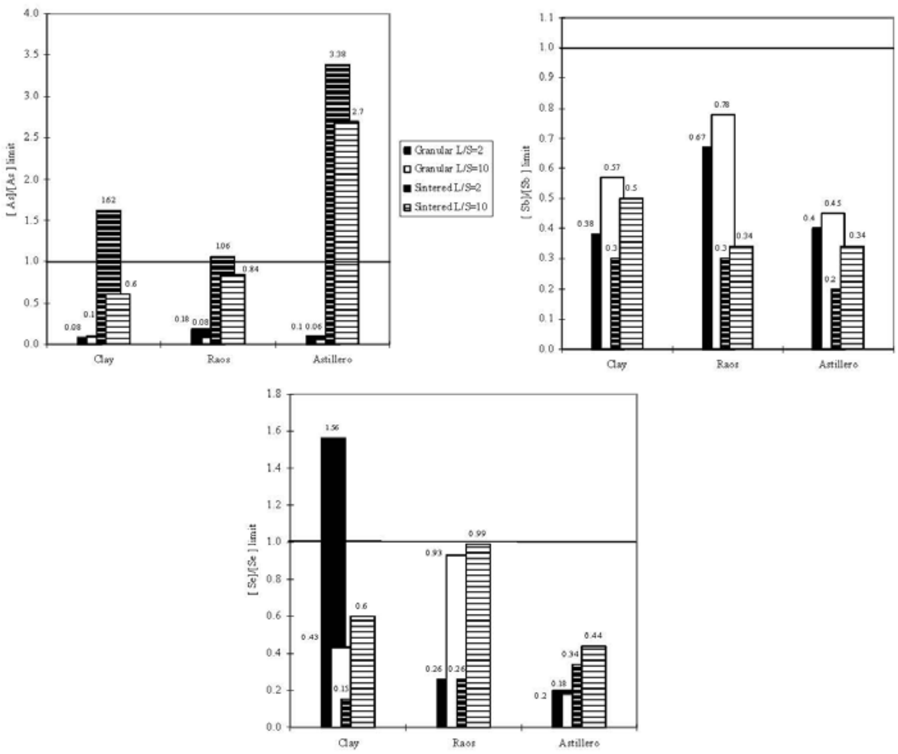

FIGURE 2 - Dimensionless concentrations of As, Se and Sb in leachates obtained in UNE-EN 12457 test for clay, Astillero and Raos samples.

Figure 3 plots the results obtained in the analysis of the leachates for fluoride, chloride and sulphate.

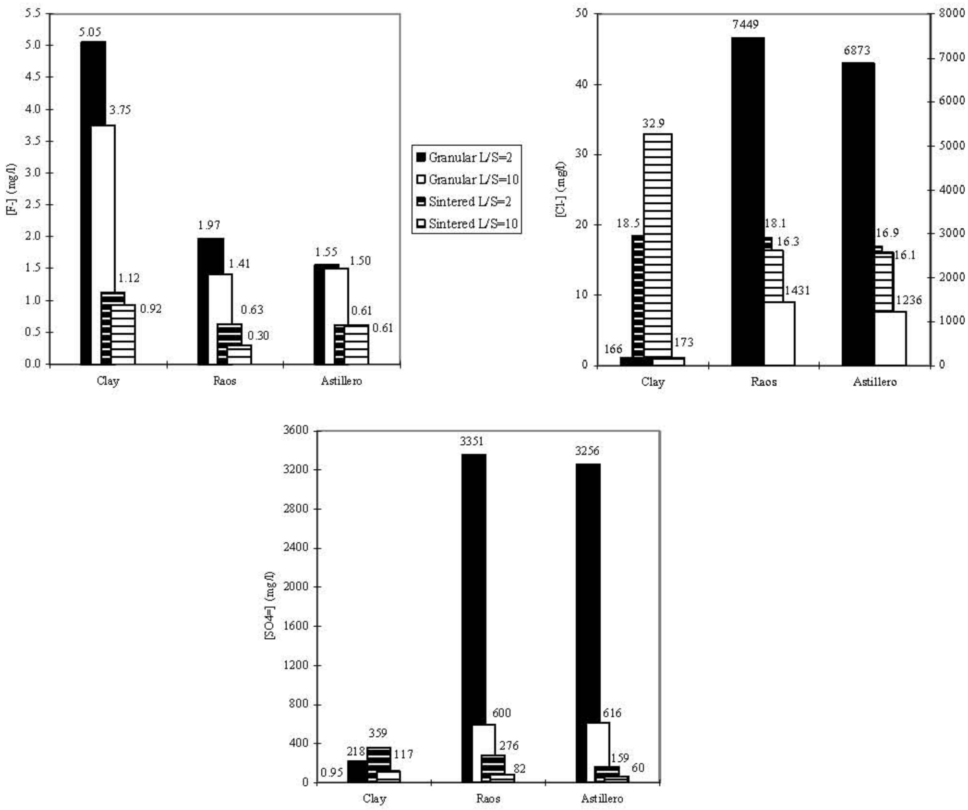

FIGURE 3 - Concentrations of fluoride, chloride and sulphate in leachates obtained in UNE-EN 12457 test for clay, Astillero and Raos (Chloride concentrations in granular samples are referred to the secondary axis).

If the relation between L/S ratio and mobility of anions is analyzed, it can be seen that a higher L/S results in a lower leaching of fluoride, chloride and sulphate in tests performed both in granular and sintered samples.

The firing process strongly influences the leaching of these anionic species in sintered bodies, decreasing the transfer of these anions into the liquid phase, except for sulphate in clay. This latter observation can be attributed to the formation of sulphates (efflorescence potential) in the clay-based product during firing by exposure to sulphurous gases coming from oxidation of insoluble sulphides in the product, and reaction of sulphates with silicates [25] However, in further studies, a gaseous release estimation will be developed due to the relation of fluoride, chloride and sulphate with HF, HCl and SOx, which are considered to be pollutants, in the Spanish Emissions and Pollutant Sources Register [26].

In order to establish the environmental risk of both granular and sintered specimens, anionic concentrations and regulatory limits from Waste Landfill Directive are compared. Both dimensionless concentration and required limits are shown in Figure 4. In sintered bodies, the normalized limit value is slightly exceeded by sulphate present in clay both in L/S=2 and 10. Nevertheless, in granular samples fluoride concentrations are above the limit in clay in L/S=2 and 10, and in sediments in L/S=10. Chloride concentrations are above the limits in clay in L/S=10, and in sediments in both liquid to solid ratios. Sulphate concentrations are above the limits in sediments in L/S=2 and 10.

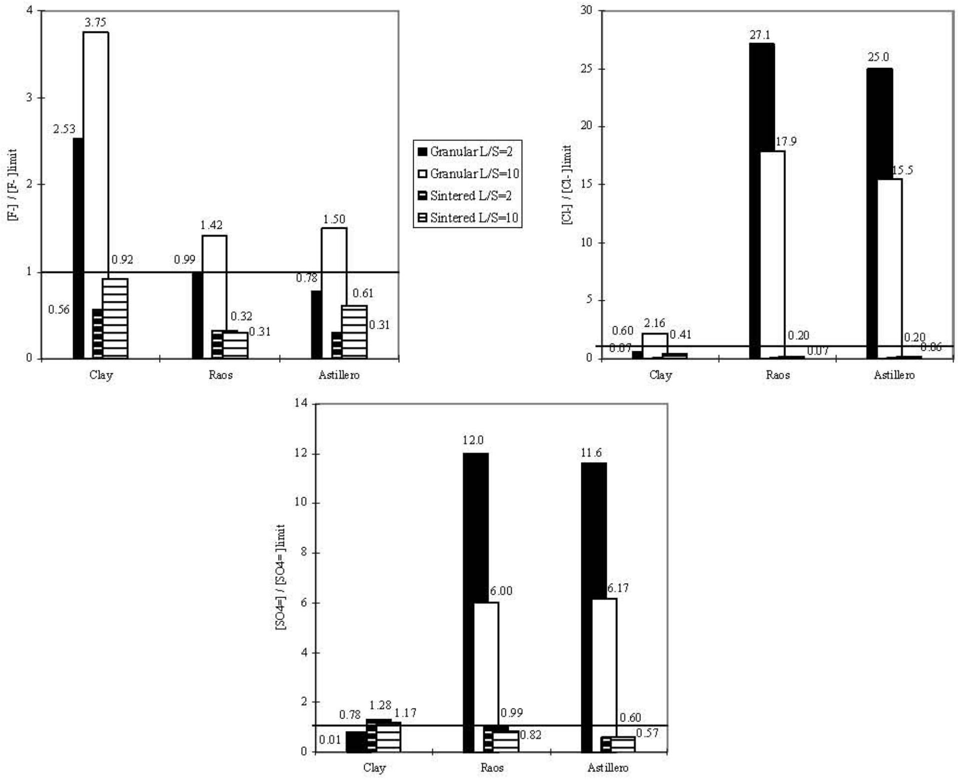

FIGURE 4 - Dimensionless concentrations of As, Se and Sb in leachates obtained in UNE-EN 12457 test for clay, Astillero and Raos sediment samples.

Identification of leaching mechanism and solubility versus availability control, from leaching tests has been carried out. Data expressed in mg/L reflect solubility control when data for different liquid to solid ratios coincide. In contrast, when leaching is controlled by species availability, this trend is the opposite. In this case, availability is the mechanism responsible for the leaching of As and Sb, both in granular or sintered samples. However, the controlling leaching mechanism of Se cannot be established due to the trend which is not clear. With regards to leaching of fluoride, chloride and sulphate, the mechanism responsible for leaching in the sintered samples is also availability. In contrast, in granular samples the trends are not clear.

CONCLUSIONS

Marine sediments from Raos and Astillero (Cantabrian Sea) can be considered to be of category II, according to Action Levels proposed by CEDEX [19], which implies medium contamination, whereas the disposal at sea is permitted for clay belonging to category I. Thus, it can be concluded that sediments need an environmental study prior to their controlled disposal into the sea.

The sintering process reduces the leaching of the metals and metalloids analyzed, except for As in all samples and Ba and Mo only in Raos sample. This fact can be due to the reactions produced during sintering, which decrease the release of these species into the liquid phase.

The leaching of fluoride, chloride and sulphate is lower in sintered specimens than in granular samples, except for sulphate in clay. Due to the relevance of these species in gaseous emissions, a gaseous release estimation will be developed in further studies.

The comparison between the regulatory limits collected in Waste Landfill Directive 2003/33/CE and the concentrations present in leachates shows that all sintered specimens were in compliance with European Regulatory Limits. In granular samples, fluoride, chloride and sulphate are above the required limits.

The larger the liquid to solid ratio, the lower the concentration of species in leachates. This fact reflects an apparent availability control in the leaching of As and Sb both in granular and sintered samples, while the controlling mechanism of the leaching of Se cannot be established. Besides, the leaching of fluoride, chloride and sulphate is controlled by an availability mechanism in sintered samples, while no trends in granular samples can be appreciated.

the environmental risk associated to the sintered sediments can be considered to be lower than that associated to the unfired sediments, except for As. In general, the results from contaminated marine sediments and clay are similar. Therefore, the results promote the potential application of sediments in brick manufacturing.

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