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Leaching properties of slag generated by a gasification/vitrification unit: The role of pH, particle size, contact time and cooling method used

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1. Introduction

The effective management of solid waste involves the application of various treatment methods, technologies and practices. All applied technologies and systems must ensure the protection of public health and the environment. The choice of thermal methods is mainly followed because energy can be reclaimed from waste and the volume and weight of the treated waste is significantly reduced. Thermal treatment technologies include incineration, pyrolysis, gasification, and plasma technology [1].

Plasma gasification, an emerging and promising technology, is the most common plasma process for waste treatment [2,3]. It is an advanced gasification process which is performed in an oxygenstarved environment to decompose organic input waste into its basic molecular structure. The organic waste is converted into a fuel gas that maintains all the chemical and heat energy from the waste, while the inorganic waste is converted into an inert vitrified slag [4,5].

The plasma technology can be used for the thermal treatment of almost any type of waste. There are two generic configurations of plasma gasification: configurations in which the plasma generator is external to the main waste conversion reactor and is used as a source of hot gases (this is often referred to as "plasma assisted gasification") and those in which the plasma generator (plasma torch or electrodes) is contained within the main waste conversion reac-

ABSTRACT

The environmental impact from the operation of thermal waste treatment facilities mainly originates from the air emissions, as well as the generated solid residues. The objective of this paper is to examine the slag residue generated by a demonstration plasma gasification/vitrification unit and investigate the composition, the leaching properties of the slag under different conditions, as well as the role of the cooling method used. The influence of pH, particle size and contact time on the leachability of heavy metals are discussed. The main outcome is that the vitrified slag is characterized as inert and stable and can be safely disposed at landfills or used in the construction sector. Finally, the water-cooled slag showed better resistance in relation to heavy metal leachability compared to the air-cooled slag.

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tor [6–9]. An indicative plasma assisted gasification unit is the one owned by Hitachi Metals with treatment capacity of 200 tonnes of municipal solid waste and automobile shredder residue per day in Utashinai in Japan [10].

The first attempt to apply gasification process in Greece was made by the Unit of Environmental Science & Technology of the National Technical University of Athens [11,12] with a unit in which the plasma generator is located within the waste treatment reactor.

The inorganic part of the treated waste, which is melted, drops to the bottom of the furnace and is removed from the lower part of the furnace and collected in a fire resistant pan. The slag is either poured in a slag mold to form ingots (air-cooled slag, mentioned as ACS, Photo 1) or quenched in a water tank to produce granulated slag (water cooled slag, mentioned as WCS, Photo 2). Their physical appearance is visually differentiated, as the first is bulky, while the second is in granular form. This vitrified material can be used as raw materials for cement, for road embankments, unbound foundations, bituminous mixture, etc. saving several million tons per year of natural materials and dramatically reducing landfill-related problems [13–16]. This is why the extensive study of the leaching properties of the vitrification process is of great interest [17,18].

2. Experimental

In order to examine the slag generated and the operation of the plasma gasification/vitrification unit, it was chosen to use waste input characterized by both high metal content and organic load. For this purpose ash from foundries was mixed with shredded

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Photo 1. ACS.

municipal waste in a ratio 1:1 and was used as feeding material in the gasification/vitrification system.

2.1. Slag characterization with the use of XRD analysis

The amorphous or crystalline nature of ACS and WCS was determined by X-ray diffraction (XRD). Samples were crushed to fine powder in an agate mortar and then were scanned with Cu K α radiation from $10 \le 2\zeta \le 70^\circ$ at a scanning speed of 0.3° /min, using a Siemens D5000 powder X-ray diffraction unit, operating at 30 mA and 40 kV.

2.2. Calculation of the percentage of the organic carbon contained

These experiments were conducted in order to obtain additional confirmation that the gasification process has been completed, by verifying that the carbon contained in the waste has been gasified. The procedure that was used to determine the percentage of organic carbon is the Walkley-Black Wet Combustion method. Slag samples were pulverized and the fine-granule particles were used ($d < 150 \,\mu$ m). 50 mg of pulverized sample was mixed with 10 ml of K₂Cr₂O₇ 1 N. Then, 20 ml of H₂SO₄ 98% were added and the samples were shaken for 1 min. The samples were left to cool for 30 min and then 20 ml of deionized water and 10 drops of



Photo 2. WCS in the form of granules.

Ferroin indicator were added. The samples were titrated with FeSO₄·7H₂O 0.5 N. The same procedure was also followed for the blank experiment, performed without the use of slag samples.

The percentage of the organic carbon contained in the samples is calculated according to the equation:

$$% C_{\text{org}} = \frac{(B-T) \times N \times 3 \times 1.14 \times 100}{50 \text{ mg dry sample}}$$

where *N* is the normality of $FeSO_4 \cdot 7H_2O$ solution. *T* is the ml of $FeSO_4 \cdot 7H_2O$ solution used during titration for blank experiment without slag sample. *B* is the ml of $FeSO_4 \cdot 7H_2O$ solution used during titration for experiment using slag samples.

2.3. Determination of the heavy metal content of WCS and ACS

The produced slag (both WCS and ACS) was digested with acids using the device HACH/23120-21, in order to assess its metal content. Both samples of 0.5 g were placed in a conical bottle and mixed with 4 ml of H_2SO_4 (97% purity). The bottles were put in the special digestion device, in which the temperature rose to 440 °C. After 5 min, 16.7 ml of H_2O_2 (30%) were added in the bottle and within a minute after the last drop fell, the procedure was stopped. The conical bottles were left to cool and their content was filtered under vacuum conditions. The metal content was determined using Atomic Absorption Spectrometry (AAS) (Fast Sequential Atomic Absorption Spectrometer VARIAN AA24OFS). The following equation was used in order to assess the contained metals in the initial slag samples:

$$C_{in} = C_m \left(\frac{V_m}{1000}\right) \left(\frac{1000}{0.5}\right)$$

where C_{in} is the concentration at the slag sample, C_m is the concentration measured in AAS, and V_m is the volume measured from the filtration [19].

2.4. Leachability of heavy metals

2.4.1. Toxicity characteristic leaching procedure (TCLP) test

The TCLP test is a complicated leaching procedure that simulates conditions of waste if it was to be disposed off in an ordinary sanitary landfill. The TCLP method was used so as to evaluate the amount of heavy metals which extracts from the samples under standard conditions. According to the U.S. Environmental Protection Agency [20], the ratio liquid (l)/solid (kg) is equal to 20/1 and the contact time 18 h.

2.4.2. Leachability of heavy metals according to European decision 2003/33/EC

Decision 2003/33/EC rules the check of leachability of heavy metals from nonhazardous waste in order to be characterized as inert and can be disposed safely [21]. The procedure uses the ratio of liquid (l)/solid (kg) equal to 10/1, particle diameter less than 4 mm and contact time equal to 24 h.

2.5. Evaluation of the leachability of heavy metals as function of pH and particle size

In order to evaluate the leaching of heavy metals from slag samples, batch experiments were carried out with different sized slag particles in contact with solution at different pH values. In order to check the leachability of heavy metals in extreme conditions, a range of pH between 3 and 8 (different series of experiments for pH values 3, 4, 5, 6, 7 and 8) was selected and two particle sizes were used. The first type was grained particles with diameter less than $150 \,\mu\text{m} (d < 150 \,\mu\text{m})$ and the second type particles had diameter between $150 \,\mu\text{m}$ and $2 \,\text{mm} (150 \,\mu\text{m} < d < 2 \,\text{mm})$. In order to carry

Table 1 Content of organic carbon in the samples.

Sample	Consumption of solution $FeSO_4 \cdot 7H_2O(ml)$	% C _{org}
Blank solution ACS	19.9 19.7	0.684
WCS	19.9	0

out the experiments, the slag samples were first pulverized and sieved in order to get two fractions of fine-grained particles, those with diameter less than 150 μ m ($d < 150 \mu$ m) and those with diameter between 150 μ m and 2 mm (150 μ m < d < 2 mm). At this point, it should be noted that it was really a difficult task to pulverize the slag samples. In fact, a hammer was used at first and, then, a mortar so as to achieve in getting fine-grained particles. It was decided to work with fine grained slag particles in order to have a form of slag characterized by increased leachability, due to the larger surface area in contact with the solution. This way, the measured heavy metal concentration in the leachate will represent a maximum of the leaching level. Deionized water was used as solvent, while the ratio of solid (kg) to liquid (l) was equal to 1/20. 5 g of the slag sample (the experiments were carried out for both WCS and ACS, as well as both particle sizes, $d < 150 \,\mu\text{m}$ and $150 \,\mu\text{m} < d < 2 \,\text{mm}$) were added in 100 ml of deionized water. Then, an adjustment was made so as to receive the appropriate pH values. The samples were left in room conditions (25 °C) for different time intervals. It should be noted that the pH range from 3 to 8 was chosen so as to cover the lower and upper pH limit that may be encountered in natural environment

3. Results and discussion

3.1. XRD results

The XRD pattern indicates that WCS is composed mainly of amorphous and traces of crystalline phases. Crystalline phases were identified by comparing intensities and positions of Bragg peaks with those listed in the Joint Committee on Powder Diffraction Standards (JCPDS) data files. The crystalline phases that could be identified were cristobalite (SiO₂), corundum (Al₂O₃), mayenite (Ca₁₂Al₁₄O₃₃) and iron aluminum oxide (Fe_{1.006}Al_{1.994}O₄). As a result, it is clear that in the WCS the heavy metals are mainly in the noncrystalline structure.

The XRD pattern of the ACS revealed only an amorphous phase and no crystalline structures or phases are observed.

It should be noted that the basicity (mass ratio of CaO to SiO_2) of the waste input in all cases was rather low (around 0.5) allowing high amorphous vitrification fraction even for the case of air cooled slag [22].

3.2. Calculation of the percentage of the organic carbon contained

The results for the assessment of the organic carbon contained in the two samples (ACS and WCS) are displayed in Table 1.

On the basis of the results, extra evidence is given that the gasification process of the modified unit was effective, as the percentage



Fig. 1. Comparison of the limits set by the EPA US with the metal concentrations in leachates from ACS and WCS at TCLP test.

of organic carbon contained in ACS is very little (0.684%) and in WCS nondetectable.

3.3. Determination of the heavy metal content of slag samples

The heavy metal content of the slags was determined using the digestion method with H_2O_2 and the results are shown in Table 2.

The results show that the slag samples contain at least eight different metals and the relevant content is a bit higher in the case of WCS, for which the content in Cu, Fe, Pb and Cr is considered high. It should be noted that the differences in the metal content of the generated slags are attributed to the fact that they originated from the treatment of different waste input.

3.4. Leachability of heavy metals

3.4.1. Toxicity characteristic leaching procedure (TCLP) test

The results from TCLP may give a first view of the extent of the metal leachability from the solid samples. As shown in Fig. 1, the concentrations of the metals in the leachate are far less than the concentrations contained in samples, forming in that way a first conclusion, that slag traps heavy metals. Measurements from TCLP are compared with the environmental limits that US EPA [20] has set (limits exist only for certain metals that can cause damage to human health and the environment). On the basis of this figure, it is concluded that in no case the concentration of the metal extracted exceeded the EPA limits, which consists main evidence for the stability of the vitrified slag and the fact that it can be safely disposed in the case that no market is available for this material despite its relevant potential to be utilized in construction applications, etc.

The different redox conditions can result in significant modification of the leaching potential for just certain metals that are characterized as redox sensitive ones. In general, metals like Cu, Zn and Pb are expected not to be particularly affected by the change in redox potential, while such change can either increase or decrease the leaching potential in the case of other metals. Indicatively, Fe²⁺ that is more favored under anoxic conditions is more soluble than Fe³⁺ and therefore the leaching potential is greater, while Cr³⁺ is more stable and less mobile that Cr⁶⁺ leading to lower leaching levels.

Table 2	
Concentration of the metal content of WCS and ACS.	

Sample	Concentrati	Concentration of the metals measured in WCS and ACS after digestion (mg/kg)								
	Cr	Cu	Mn	Fe	Ni	Cd	Pb	Zn		
WCS	185.26	711.48	203.28	604.85	246.80	21.44	330.79	165.82		
ACS	13.40	73.58	101.21	308.35	12.26	4.47	27.16	50.89		

Comparison of the limits set by the EPA US with the



Fig. 2. Comparison of the limits set by the European Decision 2003/33 with the actual respective metal concentrations in leachates from ACS and WCS.

3.4.2. Leachability of heavy metals according to European Decision 2003/33/EC

The results from the evaluation of the metals extracted according to the procedure of the European Decision 2003/33/EC are shown in Fig. 2. The results from the experiments were compared to the limits set by the EU. It is very important to mention that the amounts extracted are far less than those from previous methods and in few cases the concentrations measured are nondetectable. It was found that the metal concentration extracted from the samples is in every case lower than the EU limits.

3.5. Evaluation of the leachability of heavy metals as function of pH and particle size

Based on the results obtained, it was found that only some of the metals contained in the samples were extracted at detectable level. In all experimental series the concentration of Ni, Pb and Cr in the leachate obtained was not detectable. Fig. 3a–d presents the results for the leaching percentages of Cu in relation to the initial Cu content of the slag samples for the 6 different pH values.

Fig. 3a refers to WCS and particle size less than 150 µm, Fig. 3b refers to WCS and particle size between 150 µm and 2 mm, Fig. 3c refers to ACS and particle size less than 150 µm and Fig. 3d refers to ACS and particle size between $150\,\mu\text{m}$ and $2\,\text{mm}$. It can be seen that Cu leachability depends on pH, contact time and particle size. Leachates from both WCS and ACS samples have higher Cu concentration at low pH values, such as 3 and 4, whereas at pH values, such 7 and 8. Cu concentration is nondetectable. Cu concentration in leachates of large sized particles of ACS and WCS samples are considerably less than those of fine particles. Furthermore, it can be seen that Cu concentration in the leachates of large sized samples reached a maximum value and stabilized faster (in 20 days), whereas stabilization of Cu concentration in leachates of finer samples was attained after a longer period (44 days), showing a dependence on the particle size. However, in both large and small particles of ACS and WCS samples the Cu concentration extracted stabilizes faster at higher pH values (pH > 5), whereas at lower ones (pH 3 or 4), it requires more days. Specifically, Cu concentration in the leachates of fine-granule samples does not appear stabilized until the 44th day, whereas Cu concentration in leachate obtained from large particles stabilizes in 20 or less days.

Fig. 4a-d presents the relevant results for the case of Mn.

Based on Fig. 4, it is shown that there is also dependence of Mn leaching properties on contact time, pH and particle size. The time required for the stabilization of a maximum Mn concentration in leachates is also long (44 days) for the fine-granule ACS, but Mn concentration in the leachates of WCS is attained faster (20 days). It should be mentioned that at low pH values (pH < 5) the % of Mn extracted from fine grained samples of both WCS and ACS was relatively high (>35%) compared to the percentage of Mn extracted from large sized particles. The maximum percentage of Mn extracted from large sized particles of WCS was below 4%, whereas Mn concentration in the leachates of ACS with large sized particles was nondetectable. This may be attributed to the partly different structure. More specifically, ACS is totally amorphous as proved by the XRD results discussed at paragraph 3.1 and the leaching concentration of metals in crystalline slags is relatively



Fig. 3. (a-d) Cu leaching percentage from slag samples as function of contact time and pH.



Fig. 4. (a-d) Mn leaching percentages from slag samples as function of contact time and pH.

high in comparison to amorphous slags, since acid corrodes faster the crystalline phase rather than the amorphous one [23]. Also, as expected, in the case of Mn, higher pH values result in less metal leaching. The contact time required for obtaining the maximum Mn leaching concentration in some cases is relatively low (20 days and in a specific case 5 days).

Fig. 5a-d presents the relevant results for the case of Fe.

On the basis of Fig. 5, it is shown that Fe is leachable only under certain conditions and more specifically only at low pH values (pH < 5). Even for fine-granule samples, the Fe concentration in the leachates is almost nondetectable at pH values higher than 5.

Fig. 6a–d presents the relevant Cd leaching percentages and shows that Cd is characterized by a similar trend to the metals aforementioned. Furthermore, Cd concentration in the leachates of both WCS and ACS samples with large particle size was nondetectable for all pH values. The highest percent (7.6%) of Cd was extracted from ACS fine grained samples at pH 3 and stabilized after 54 days, while in WCS the highest percentage extracted from fine grained WCS at the same pH value of 3 was lower than 3%. However, in this case the Cd concentration in the leachates is not stabilized within the time interval of the experiments (64 days), showing tendency for a slight increase with time.



Fig. 5. (a-d) Fe leaching percentages from slag samples as function of contact time and pH.



Fig. 6. (a-d) Cd leaching percentages from slag samples as function of contact time and pH.

Fig. 7a–d presents the relevant results for the case of Zn. However, in the case of Zn, even higher pH values result in considerable Zn concentrations in leachates for both samples and both particle sizes. As for WCS, Zn concentration in the leachates stabilizes in 44 days. The samples with pH values 7 and 8 where the Zn concentration stabilizes in 11 days constitute exceptions. The highest metal extraction for the WCS was measured for pH value 3 (around 19%). Similar were the results for ACS. Specifically, in fine-granule samples the leaching concentration stabilized in 44 days for pH value 3, reaching the maximum value of Zn extraction, 48%. In the samples consisting of larger particles, at pH values 3 and 4, the Zn concentration in the leachates reached maximum level from the 5th day (maximum % Zn extraction was 8% for WCS and 20% for ACS), whereas in the rest samples in 11 days.

The different behavior of Cd compared to Zn in the $150 \,\mu\text{m} < d < 2 \,\text{mm}$ fractions could be explained by the different metal forms within the slags. Zn can be found into spinel crystals, the glassy matrix itself, as well as in metallic form, while



Fig. 7. (a-d) Zn leaching percentages from slag samples as function of contact time and pH.

the available information about the Cd speciation is rather limited [24].

The leaching experiment series under the specific conditions described (pH, particle size, contact time) revealed that the leaching percentages of the metals vary in the range:

- Cu: 0.12-26.66%.
- Mn: 0.99-49.21%.
- Fe: 0.026-33.14%.
- Cd: 1.72-7.61%.
- Zn: 0.48-48.38%.

In general, the metal concentrations in the leachates are higher at lower pH values and tend to minimize when reaching the values of 7 and 8. The extremely low metal leaching in the case of pH values of 7 and 8 can also be attributed to precipitation that occurs at these pH values. In addition, the concentration of the metals in leachates reaches maximum values after contact time of several days.

According to bibliography, the difference in leachability for particles with different diameter is more obvious between the finegrained samples and those which have diameter greater than 4 mm. However, this type of experiments attempted to show this correlation even for the particles which do not differ so much in size. It is important to mention that the samples with larger particles lead to lower metal leaching concentrations than those of smaller particles.

Moreover, the leaching properties of the slag seem to be influenced by the cooling method used. More specifically, experimental results show that metal leaching is enhanced in ACS. Water quenching can enhance more uniform distribution of the glassy amorphous phase. The experimental results show that the effect of the cooling method on the leachability of metals is more pronounced for the case of Zn and Cu and relatively less for Mn.

Even though samples and especially WCS contained significant amounts of Ni, Pb and Cr, leaching of these metals was not detectable under all experimental conditions indicating that vitrification greatly minimizes the leaching amounts. For the case of Pb, this can be explained by the affinity to bind to solids. On the other hand, Zn and Cu seem to extract in high concentrations, while Cd had significantly lower leaching concentrations. Nevertheless, the slag produced from the treatment of waste with high inorganic load is characterized by generally low leaching properties.

4. Conclusions

In this work, the slag generated from a gasification/vitrification unit is assessed in relation to its leaching behavior. Two slag types were obtained, the air cooled slag (ACS) and the water cooled slag (WCS). Both ACS and WCS produced are mainly composed of amorphous glassy phases. The results obtained show that the vitrified slag is characterized as stable and inert and can be safely disposed complying with both TCLP and Decision 2003/33/EC requirements. In fact, the vitrified slag appears to have similar leachability with road construction materials, fact which strengthens the opinion that slag can be used in several applications. It has to be noted that limit conditions were used in the experiments in order to determine the maximum metals extracted from the slag samples. For this reason, extremely small particles of slag were used and experiments also included very acidic environment (pH values 3 and 4), as well long contact time (until 64 days).

The leaching experiments performed led us to the major conclusion that the leaching behavior depends on pH value, particle size and cooling method of slag. Rapid cooling of the generated slag improves stabilization of metals in the glass phase and enhances acid resistance of slag. The leaching behavior of the slag samples with respect to Cu, Mn, Fe, Cd, Zn, Ni, Pb and Cr showed that the vitrification process immobilized Cr, Pb and Ni in the glass matrix, significantly reduced Cd leaching, while for Cu, Mn, Fe and Zn the leaching amounts at low pH values were high.

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