

UTILIZATION OF CONCRETE AND ASH WASTE IN GEOTECHNICAL CONSTRUCTION

Legislation, methods and analysis
requirements in Finland and Spain

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ABSTRACT

The construction sector generates a larger amount of waste each year. That is why new laws and rules, which regulate the management of such waste, are appearing continuously, in order to end the problem that these wastes cause by throwing into the environment, without any control or regulation.

One of the main objectives of this project was to study both Finnish and Spanish legislation as well as the regulations, methods and treatments necessary for the use of these wastes as new by-products or raw materials.

To determine if a particular type of waste is suitable for reuse, three samples of bottom ash provided by the company Lahti Energy, were treated by a leaching behaviour test. This test makes it possible to obtain some liquid samples from the previous solids. Then these liquid samples have been analysed through an ecotoxicological pilot test with *Artemia Salina* and in addition, a photometer was also used to determine the concentrations of chromium and copper present in the samples.

The results of the tests indicate that although the three samples have some type of harmful substance for the environment, only sample number 2 exceeds the limits established by the Government decree (591/2006) of Finland and therefore cannot be reused. Also, it has been verified that the ecotoxicological test with *Artemia Salina* is a good option to determine the toxicity of the samples to be analysed.

Key words: leaching behaviour test, ecotoxicological test, *Artemia Salina*, waste, bottom ash, reuse

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RESUMEN

El sector de la construcción, genera cada año una cantidad más elevada de residuos. Es por ello que nuevos tratamientos, leyes y normativas, que regulan la gestión de dichos residuos, van apareciendo día a día con la finalidad de acabar con el problema que supone que estos desechos sean arrojados al medio ambiente, sin ningún tipo de control o regulación.

Uno de los objetivos principales de este proyecto es hacer un estudio tanto de la legislación Finlandesa como de la Española así como a su vez de las normativas, métodos y tratamientos necesarios para el uso de estos residuos como nuevos subproductos o materias primas.

Para poder averiguar si una cantidad de residuo es apta o no para su reutilización, tres muestras de cenizas y escombros proporcionadas por la empresa Lahti Energy, han sido tratadas mediante un test de lixiviación, que ha permitido obtener unas muestras líquidas que posteriormente han sido analizadas mediante un estudio ecotoxicológico piloto con *Artemia Salina*. Además también se ha usado un fotómetro para determinar las concentraciones de cromo y cobre presentes en las muestras.

Una vez realizados dichos ensayos/ tests, los resultados indican que aunque las tres muestras presentan algún tipo de substancia nociva para el medio ambiente, sólo la muestra número 2, supera los límites establecidos por el decreto gubernamental (591/2006) de Finlandia y por tanto no podría ser reutilizada. Además se ha comprobado, que el ensayo ecotoxicológico con *Artemia Salina*, sí que es una buena opción para determinar la toxicidad de las muestras a analizar

Palabras clave: test ecotoxicológico, *Artemia Salina*, test de lixiviación, residuos, cenizas, reutilizar

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LIST OF ABBREVIATIONS

As	Arsenic
Ba	Barium
Cd	Cadmium
C&D	Construction and Demolition waste
Cl	Chloride
Cr	Chromium
Cr ^{III}	Trivalent chromium
Cr ^{VI}	Hexavalent chromium
Co	Cobalt
Cu	Copper
DOC	Dissolved Organic Carbon
EU	European Union
F	Fluoride
HCl	Hydrochloric acid
HF	Hydrogen Fluoride
Hg	MErcury
Mn	Manganese
Mo	Molybdenum
Ni	Nickel

Pb	Lead
SO ₂	Sulfur dioxide
SO ₄	Sulphate
SRF	Solid Recovered Fuel
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
V	Vanadium
Zn	Zinc

1 INTRODUCTION

According to the Finnish legislation, waste means any substance or object which the holder discards, intends to discard or is required to discard (Finnish Waste Act 646/2011, 5 §).

Nowadays, however, it has been seen that these substances or objects may have a second life, either as a by-product or raw material (Manskinen 2013, 3). With the aim of finding out what kind of laws, decrees and treatments are necessary for the reuse of such waste, this thesis includes a study of the legislation in both Finland and Spain.

This study is focused on the concrete and ash waste utilization in geotechnical construction. The boom experienced in the construction sector, has involved the generation of significant amounts of construction and demolition waste (C&D) and due to the lack of planning much of it has been deposited in landfills or disposed of in an uncontrolled way. This growing generation of waste implies a problem for many countries, not only because it is wasted energy and potentially reusable, recyclable or valuable material but because the environment is being affected in a very negative way. That is why some of these countries have begun to solve this problem by creating laws, regulations or new methods of recycling.

Although Finland and Spain belong to the EU, they are not governed by the same rules, so it is interesting to see how they differ or where they coincide and also see what things could be improved in each of these two countries.

This thesis informs about the laws and also makes a small study of what kind of treatments and standards are necessary for this reuse. The main focus is on leaching behaviour tests, which permit to obtain some liquid samples from the previous solids of ash and concrete, which then could be evaluated and characterized.

One of the ways used for the analysis of these liquid samples, has been the use of a photometer, which allows determining the concentration of

some harmful substances present in the sample such as copper or chromium.

The reuse of these products is not easy since most of them contain multiple harmful substances that could cause serious damage to the environment. It is for this reason that the main objective of this project, besides making a theoretical study of the laws as has been said before, is to estimate the toxicity of three different samples of bottom ash waste provided by the company Lahti Energy through an ecotoxicological pilot study with *Artemia Salina* (a kind of crustacean which lives in salt water and has simple organism and a primitive nervous system, which make them suitable for toxicological tests). This type of tests relates to the mortality of these organisms with the toxicity of the samples to be analyzed.

2 LEGISLATION

2.1 Finnish environmental legislation on waste utilisation

Finnish waste legislation is largely based on EU legislation, and covers almost all wastes except certain special types of waste such as radioactive wastes, which are covered by separate laws (Finnish Waste Act 646/2011, 3 §). This legislation in some cases also includes stricter standards and limits than those applied in the EU as a whole and some issues related to wastes that have not yet been covered by EU legislation.

The main Finnish laws that contain the information about the reuse and recovery of the concrete and ash waste are: Environmental Protection Act (86/2000), Government Decree on waste incineration (151/2013), Government Decree concerning the recovery of certain wastes in earth construction (591/2006), Waste Act (646/2011) and Waste Decree (179/2012).

According to the Finnish legislation waste means any substance or object, which the holder discards, intends to discard or is required to discard (Finnish Waste Act 646/2011, 5 §). However, this waste could also be seen as a by-product or raw material (Manskinen 2013, 3). So that the reuse of these wastes could be possible, there are a few requirements that must be fulfilled depending on the type of waste you are working with (Mroueh & Wahlström 2002).

2.1.1 Concrete and ash waste utilization in geotechnical construction

According to the Environmental Protection Act (86/2000), which applies to all activities that lead or may lead to environmental pollution, no permit is needed for the use of wastes in earth construction. However, the process of recovery have to be notify to the environmental authorities responsible for waste management (Finnish Government Decree 403/2009, Annex 1).

Concerning the recovery of certain wastes in earth construction, there is the Government Decree (591/2006), which is only applicable to institutional or commercial recovery of wastes (it cannot be applied in important groundwater areas for building public roads, streets, bicycle lanes, parking areas, railway yards, etc).

In addition to the requisites in the Environmental Protection Act (86/2000) that have been explained before, there are a few requirements more that must be fulfilled. First of all, the limits of content and leaching of harmful substances shown in Tables 1 and 2 cannot be exceeded (Government decree 591/2006, Annex 1).

TABLE 1. Limits on the content of any harmful substance in the case of concrete chippings (Government decree concerning the recovery of certain wastes in earth construction 591/2006)

Harmful substance	Limit value, mg/kg dry substance Basic characterisations			Limit value, mg/kg dry substance Quality control investigation		
	Content	Leaching (L/S=10l/kg) Covered structure	Leaching (L/S=10l/kg) Paved structure	Content	Leaching (L/S=10l/kg) Covered structure	Leaching (L/S=10l/kg) Paved structure
PCB	1,0			1.0		
PAH	20					
TOC	30000					
DOC		500	500			
Antimony (Sb)		0.06	0.06			
Arsenic (As)	50	0,5	0.5	50		
Barium (Ba)		20	20			
Cadmium (Ca)	10	0.02	0.02	10	0.02	0.02
Chromium (Cr)	400	0.5	0.5	400	0.5	0.5
Copper (Cu)	400	2.0	2.0	400	2.0	2.0
Mercury (Hg)		0.01	0.01			
Lead (Pb)	300	0.5	0.5	300	0.5	0.5
Molybdenum (Mo)		0.5	0.5			
Nickel (Ni)		0.4	0.4			
Vanadium (V)		2.0	2.0			
Zinc (Zi)	700	4.0	4.0	700		
Selenium (Se)		0.1	0.1			
Fluoride (F ⁻)		10	10			
Sulphate (SO ²⁻⁴)		1000	3000		1000	3000
Chloride (Cl ⁻)		800	800			

TABLE 2. Limits on the content of any harmful substance in the case of fly ashes (Government decree concerning the recovery of certain wastes in earth construction 591/2006)

Harmful substance	Limit value, mg/kg dry substance Basic characterisations			Limit value, mg/kg dry substance Quality control investigation		
	Content	Leaching (L/S=10l/kg) Covered structure	Leaching (L/S=10l/kg) Paved structure	Content	Leaching (L/S=10l/kg) Covered structure	Leaching (L/S=10l/kg) Paved structure
PCB	1.0					
PAH	20					
DOC		500	500			
Antimony (Sb)		0.06	0.18			
Arsenic (As)	50	0.5	1.5	50		
Barium (Ba)	3000	20	60	3000		
Cadmium (Ca)	15	0.04	0.04	15		
Chrome (Cr)	400	0.5	3.0	400	0.5	3.0
Copper (Cu)	400	2.0	6.0	400		
Mercury (Hg)		0.01	0.01			
Lead (Pb)	300	0.5	1.5	300	0.5	1.5
Molybdenum (Mo)	50	0.5	6.0	50	0.5	6.0
Nickel (Ni)		0.4	1.2			
Vanadium (V)	400	2.0	3.0	400	2.0	3.0
Zinc (Zi)	2000	4.0	12	2000		
Selenium (Se)		0.1	0.5		0.1	0.5
Fluoride (F ⁻)		10	50		10	50
Sulphate (SO ²⁻⁴)		1000	10 000		1000	10 000
Chloride (Cl ⁻)		800	2 400		800	2400

Furthermore, it is necessary to know that if different wastes are mixed to improve technical characteristics or to level the soil structure and enhance its bearing capacity and durability, the maximum thickness of the structure containing waste is 150 cm, so it is important to be accurate with the quantity of waste that is used (Government decree concerning the recovery of certain wastes in earth construction 591/2006, 3 §). This structure has to be covered or paved and as is referred to in Chapter 1, section 4 of the Water Act (264/1961) shall not be in contact with

groundwater. Also, 30 meters is the minimum distance at which a structure containing waste could be situated from any other source of water intended for human consumption.

Finally, it must be taken into account that the temporary storage of waste and any other activities at the recovery site should not cause harm to health and the environment. The general principles for waste quality control are summarised in standards SFS-EN 12457-1/4, CEN / TS 14405, CEN / TS 14429, CEN / TS 14997.

2.1.2 Ash waste utilization as a fertiliser

A by-product of an industrial or processing plant that can be used as an ash fertiliser is a product, which promotes plant growth, mainly based on the amount of nutrients usable to plants present in the by-product (Decree of the Ministry of Agriculture and Forestry on Fertiliser Products 24/11, Annex 1).

The decree that regulates the requirements for the type designation list and the requirements for quality, marking, packaging, transporting, storage, usage and other requirements and the raw materials used in fertiliser products is "*Decree of the Ministry of Agriculture and Forestry on Fertiliser Products 24/11*". According to the decree, ashes produced in the manufacture of wood, peat or agro biomass as well as ash of animal origin may be used as a fertiliser provided that ash is treated in such a way that dusting is minimised. The decree establishes the maximum amount of some harmful substance content in ashes (Table 3).

TABLE 3. Maximum amount of some harmful substances in ashes
(Decree of the Ministry of Agriculture and Forestry on Fertiliser Products
24/11, Annex 1)

Harmful substance	Maximum concentration in agriculture and horticulture (mg/kg dm)	Maximum concentration in forestry (mg/kg dm)
Arsenic (As)	25	40
Cadmium (Cd)	1,5	25
Chromium (Cr)	300	300
Copper (Cu)	600	700
Mercury (Hg)	1,0	1,0
Lead (Pb)	100	150
Nickel (Ni)	100	150
Zinc (Zn)	1500	4500

In addition to this table, the decree of the Ministry of Agriculture and Forestry on Fertiliser Products (24/11) also sets out that the content of primary and secondary nutrient content must be declared if it exceeds 0.3 % of dry matter and that the minimum values of nutrient contents of ash fertiliser used in forest are 2.0 % for potassium (K) + phosphorus (P), and 6.0 % for calcium (Ca).

2.2 Spanish legislation

Spain produces about 40 million tonnes of concrete and ash waste and represents one of the countries of the European Union that dedicates less resources to the reuse or recycling of this waste (less than 5 %), this means that more than 95 % of the waste ends up in landfills (National

Construction Waste Plan and Demolition 2001-2006, 1 §). To correct and control this situation, some regulations and laws have been approved.

Some of the most important are:

- **Waste Act 22/2011**
- **Royal Decree 105/2008:** Regulates the production and management of construction and demolition waste
- **Law 10/1998:** Prevents the production of waste, establishes the legal regime of their production and manages and promotes the reuse, recycling and utilization of the wastes
- **Royal Decree 653/2003:** Waste incineration
- **Royal Decree 1088/1992:** Prevention and reduction of environmental pollution produced by asbestos
- **Decree 1217/1997:** Incineration of hazardous waste and modification of Royal Decree 1088/1992
- **National Construction Waste Plan and Demolition (PNRCD) 2001-2006**

The main objective of the law (10/1998) is to prevent and reduce the production of waste. For this it promotes and manages the recycling and the reuse of those wastes and set out other forms of recovery.

According to this law, a waste is any substance or object from which its holder is detached or is obliged to discard. But not all the wastes that are known are contemplated in the previous decrees. Exists the exception of radioactive wastes and wastewater that are regulated with different laws.

The decree (10/1998) says that if the owners of the waste can't manage them by themselves, they must deliver the waste to a specialized manager, who eliminates or reuses them. The owner could participate in the process and is obliged to pay the corresponding management costs.

Furthermore, this law establishes the obligation to prepare and approve a plan (National Waste Plan), which will be elaborated by all the autonomous communities of Spain. The main points of this plan, like in the

law (10/1998) is the reduction, reuse, recycling, other forms of recovery and disposal of the wastes, as well as the form to achieve that things and the funding system that is needed to do it. This plan should be review every 4 years and includes the possibility of making the modifications that are deemed convenient.

According to the current plan, the volume of construction and demolition waste, ranges from 2 to 3 kilograms per habitant per day and the percentage of this type of waste that is currently reused or recycled in Spain is less than 5 %. So the National Plan, targets a rate of reuse or recycling of at least 60 %. In order to achieve this target, the Public Administrations must encourage the creation of recycling infrastructures by the private sector.

The plan must follow the principle of hierarchy established by Waste Act (22/2011) which is based on the following steps: Prevention, preparation for reuse, recycling, another type of valuation and elimination. In all this process is compulsory to take the necessary measures to ensure that waste management is carried out without endangering human health or the environment.

2.2.1 Reuse of concrete and ash waste

Although there are multiple treatments for waste that are generated in construction, reuse of waste in new construction is the best option.

The main advantage of this process is that the waste becomes the new building material and therefore the contamination of the environment is avoided. If this type of treatment is used, it's good to know that exists two forms of reuse. The first one is the reuse of wastes in the same construction and the second one is the reuse of concrete and ash waste in a different construction.

In the first option it's only necessary to select and clean the waste that are going to be used, but in the second one, the material (the concrete and ash waste) has to be transported so other regulations have to be

contemplated. For example in the case of the use of ash, a permit is needed to ensure that the emission limits are not exceeded. These limits are shown in Table 4. (Royal Decree 653/2003, Annex 2.)

TABLE 4. Emissions limits of some harmful substances (Royal Decree 653/2003, Annex 2)

Harmful substance	Concentration (mg/m ³)
HCl	10
HF	1
SO ₂	50
TOC	10
Cd	0,05
Tl	0,05
Hg	0,05
Sb	0,5
As	0,5
Pb	0,5
Cr	0,5
Co	0,5
Cu	0,5
Mn	0,5
Ni	0,5

2.2.2 Recycling of concrete and ash waste

In the case of recycling, the waste must be converted into a new product, which will subsequently be used as the raw material for a new construction. Therefore consists of take advantage of the materials contained in the waste, for a later use in other issues. (Law 10/1998, 2 §.)

Technological centres, research centres, universities and technological companies in the last years have developed some new technologies of recycling, that have open new perspectives and possibilities in this field. Something that has also increased the use of this type of recycling techniques has been the increase of the cost of the discharge or its prohibition. Even so, in Spain the debris still represents a problem, because almost 75-80 % of the total construction and demolition waste it's not recycled. (National Construction Waste Plan and Demolition (PNRCD) 2001-2006, Annex 1.)

The process of recycling it's quite simple. First of all, normally the waste mix is separated manually with the aim of eliminating the larger residues that cannot be used in the subsequent recycling process. Then the waste mix is grinded and subjected to a magnetic separation, which will remove the small-unwanted particles. Finally the mixture is introduced in different machines until the different substances to be recycled are obtained. (Residuos de construcción y demolición 2007.)

Concrete and ash waste could be utilised as a new concrete or also this type of waste could be utilised in urban furniture, civil constructions, new roads, slabs, beams or pipes. If this it's not possible, they should be taken to a dumping site, but this alternative represent the most important ecological impact so it would be convenient to avoid it.

2.3 Comparison of Finnish and Spanish legislation

Although both Finland and Spain belong to the European Union, are two countries totally opposed in the field of recycling and reuse of waste. But

to find the causes of these differences, first of all the situation in Europe has to be analysed (Figure 1).

PNRCD (2001-2006)			
<i>Generación y reciclado de RCDs</i>			
Estado miembro	Escombros — M Tm	Porcentaje reutilizado o reciclado	Porcentaje vertido o incinerado
Alemania	59	17	83
Reino Unido	30	45	55
Francia	24	15	85
Italia	20	9	91
España	13	<5	>95
Holanda	11	90	10
Bélgica	7	87	13
Austria	5	41	59
Portugal	3	<5	>95
Dinamarca	3	81	19
Grecia	2	<5	>95
Suecia	2	21	79
Finlandia	1	45	55
Irlanda	1	<5	>95
Luxemburgo	0	n/a	n/a
Total EU-15	180	28	72

Fuente: Construction and demolition waste management practices, and their economic impacts. CE. Symonds & Ass, febrero, 1999.

FIGURE 1. Generation and recycling of construction and demolition waste in some countries in EU (National Construction Waste Plan and Demolition (PNRCD) 2001-2006, Annex 1)

Construction and demolition waste is the most abundant waste in the EU. European Commission legislation requires that at least the 70 % (measured by weight) of non-hazardous waste should be recovered by 2020.

In the National Construction Waste Plan And Demolition (PNRCD) 2001-2006, there is a table about the generation and recycling of construction waste in some of the countries of the European Union. As can be seen in Figure 1, the countries that generate more waste are Germany, UK and

France and the countries that produce less waste are Finland, Sweden and Ireland. Even so, most of these countries that generate more waste are at the same time, the countries that recycle the most.

For example UK, that generates approximately 30 MTm, also recycles the 45 % of the waste. So the process of recycling makes up for the high production of waste.

To analyse the situation in Europe, 5 groups of countries could be created according to their recycling range in these last years:

1. **Netherland, Belgium and Denmark:** These three countries recycle almost the 90 % of the concrete and ash waste. Netherland and Belgium, also recycle the 100 % of pavement from road construction waste due to the difficulty of find sites for landfill and the difficulty to obtain raw materials.
2. **Finland, Austria and UK:** The range percentage of recycling in these countries is between 40-45 %. The factors that have motivated the recycling and the reuse in these countries have been the new legislation, which force them to do a planning and control of the demolitions as well as new taxes on the dumping of waste.
3. **Sweden, Germany and France:** Recycle between 15-20 % of construction waste. The low rate in Germany is due to they use other treatments like is the crushed of residues.
4. **Italy and Ireland:** These two countries have a low percentage, they are between 6-9 %, but they are also working in some political strategies to increase this ratio.
5. **Portugal, Greece and Spain:** In the last positions, are finded Portugal, Spain and Greece; these countries do not exceed the 5 % of recycling or reuse of the wastes of the construction generated by the country.

Once analysed the sector of recycling and reuse in Europe have been analysed, it's possible to compare the situation in Finland and Spain.

The first one generates less waste and also recycles more than Spain. A reason to this problem is that in Spain, the legislation is not as tight as in Finland, for example the cost of depositing waste in a landfill is lower and the penalties that the state can impose for default on the legislation either do not exist or are actually low. Also in Spain the raw materials are easy to achieve, so part of the country prefer to pay for new material than reuse them. Quite the opposite than in Finland, whose legislation is tighter, and the taxes and penalties are higher. Also in Finland, waste management is better and more regulated by different laws and special treatments with its pertinent standard as it has been seen in Section 2.1 of this thesis. However, both countries must increase their recycling policies and improve their reuse processes with the aim of reducing the country's pollution rate.

3 SAMPLING

3.1 Origin of the samples – Lahti Energy power plant

Lahti Energy is an energy plant located in Lahti and established in 1907. This company has provided the samples for the practical part of this thesis.

Although the company was founded more than 100 years ago, it has grown considerably, adapting to the changes and new technologies.

The main objective of Lahti Energy is to generate and provide district heating and electricity to the customers. For that energy production, the company combines heat and power generation, utilizing a variety of energy sources in the production process.

Since 1998, Lahti Energy has gasified over a million tons of solid recovered fuels and wood in co-gasification, achieving a reduction of nearly 700,000 tons of the use of coal.

An example of these advances and good results is that the company has expanded their enterprise by building a second plant, Lahti Energy's Kymijärvi II power plant, which is the first gasification power plant in the world to efficiently generate electricity and district heat from Solid Recovered Fuel (SRF). The raw material of SRF is energy-containing waste. At Lahti Energy's power plant, the SRF is gasified, the gas is then purified and the resulting clean ecogas is combusted in an ordinary natural gas boiler (Figure 2).



FIGURE 2. Lahti Energy's Kymijärvi II power plant (Lahti Energy 2011)

3.2 Description of the samples

Lahti Energy has provided three different samples for analysis (Table 5). They have supplied for this thesis two bottom ash samples and one gasifier bottom ash sample. These three samples have been analysed by testing laboratory and the results are shown in Table 6 and Appendix 1.

TABLE 5. Names of the samples provided by Lahti Energy

SAMPLE 1	Power plant 1, bottom ash (<i>Fuel: Coal</i>)
SAMPLE 2	Power plant 2, gasifier bottom ash (<i>Fuel: SRF and recycled wood</i>)
SAMPLE 3	Process heating plant, bottom ash (<i>Fuel: Biomass</i>)

TABLE 6. Values of pH and dry matter percentage from the samples of Lahti Energy

	Dry matter percentage	pH value
Power plant 1, bottom ash	69.2%	11.7
Power plant 2, gasifier bottom ash	100.0%	12.1
Process heating plant, bottom ash	99.6%	11.5

Some of these results will be compared with those obtained in the practical part of this thesis, but for the moment they can help to determine if the samples of concrete and ash waste are suitable or not for reuse in geotechnical construction. For that, the values provided by a testing laboratory are going to be compared with the limits that are established in the Government Decree (591/2006).

Comparing Table 2 with Appendix 1, all the concentrations in the samples investigated are lower than the limit values of the Government Decree (591/2006), with the exception of sample 2 whose concentrations of lead (Pb), copper (Cu), antimony (Sb) and barium (Ba) are higher than the limit values. Lead, antimony and barium are high for the limit value of the covered structure, but copper is high for both covered and paved structure. Even so, solubilities are mainly low, therefore the reuse is possible by environmental permitting.

4 METHODS OF ANALYSIS

As has been seen in the legislation there are multiple methods of analysis to determine if the residue to be reused in earth construction exceeds the limits of concentration of harmful substance and therefore needs to be disposed of as an inert-waste, non-hazardous waste, or hazardous waste landfill.

In this case, it is not going to be possible to make a proper analysis to determine these concentrations, but it has been decided to use an extraction procedure according to SFS-EN 12457-3 to obtain some liquid samples and then prove their toxicity with a ecotoxicological test with *Artemia Salina*.

The concentration of chromium and copper will also be measured with Palintest photometer.

4.1 Leaching behaviour test

Before discussing the different types of leaching behaviour tests it is important to clarify what leaching is.

Leaching is understood as the process of a material releasing components (inorganic or organic pollutants) from the solid phase to the liquid phase when the material comes into contact with some liquid. This release of components is due to the dissolution of the mineral in the liquid, to adsorption / desorption processes or to complex processes affected by pH, redox potential, dissolved organic matter and biological activity. According to this definition, it can be said that leaching is a universal process. That means that whenever someone puts any material in contact with water, it will detach the components of its surface or its interior to the water, depending on its porosity. (Ylijoki, Juvankoski, Kaartinen, Merta, Mroueh, Mäkinen, Niemeläinen, Punkkinen & Wahlström, 2015.)

Once this concept has been clarified, it is possible to focus on the leaching behaviour tests. The main purpose of the different leaching tests is to

determine the concentrations of the contaminants, under the parameters determined by each test.

4.1.1 Types of leaching behaviour test

Evaluating and characterizing the environmental impact caused by some waste or materials requires different methods. A single technique is not enough to identify, quantify and characterize the components of the sample that is going to be analysed. So sometimes, a combination of two or three of them is needed to make a proper evaluation.

When this kind of tests are done, some parameters play an important role and they could affect the sample:

- The pH of leaching and the neutralizing capacity of the material.
- Particle size or area exposed to contact with the leaching liquid.
- Composition of the leachant.
- Relationship between the volume of leachate and the mass of material (L/S, expressed usually in l/kg).
- Time of contact between liquid and solid.

Other things to keep in mind is that there are two modes of operation, continuous, the liquid flows constantly through the solid, or discontinuous, the liquid and the solid remain in contact for a certain time in a closed system, and that the final liquid/solid ratio can be achieved in one or several stages. Following these requirements there are three types of characterization tests, normalized by standards CEN/TS 14405, CEN/TS 14429, CEN/TS 14997 and SFS-EN 12457/1-4.

4.1.2 Percolation test or column test

According to the CEN/TS 14405 in this test an upward flow is passed through a granular material (particle size <4mm) packed in a column of fixed dimensions. The leaching is carried out with deionized water at its natural pH (between 5 and 7.5) so that the material imposes its pH on the

leached solution. Seven fractions of eluate are collected to a cumulative total L/S of 10 l/kg. The L/S ratios of the different leachates are: two at 0.1 l/kg, one at 0.3 l/kg, 0.5 l/kg, 1 l/kg, 3 l/kg and 5 l/kg. The test is practicable in many granular materials, except for those with low permeability, such as clay soils or sedimentation.

4.1.3 pH-dependence test

The test consists of obtaining parallels leached from a material with an L / S ratio of 10 l/kg for 48 hours in a series of predefined pH values. The range of pH values to be comprised is between 4 and 12 inclusive and the difference between two consecutive pH values can't exceed 1,5.

In this case of test, we can operate in two different ways. One option is according to CEN/TS 14429 with an initial addition of acid or base and the other one following the CEN/TS 14997 having a continuous pH control.

4.1.4 Batch test

In these tests the granular material is exposed to a leachate, deionized water without pH adjustment, for a period of time (24 h) to establish equilibrium.

Thanks to these tests, it can be checked whether or not a material fulfil the regulations and the expected behaviour of a material or group of reference material. Besides, they are usually simple and easy tests, which imply both practical and economic advantages.

The batch tests are regulated by the standards EN 12457/1-4 (Characterization of waste – Leaching – Compliance test for leaching of granular waste materials and sludges). These documents have been prepared by Technical Committee CEN /TC 292, "Characterisation of waste" and all of them, are applicable to any waste from extractive industries.

- **SFS-EN 12457-1:** One stage batch test at a liquid to solid ratio of 2 l/kg with particle size below 4 mm (without or with size reduction).
- **SFS-EN 12457-2:** One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 4 mm (without or with size reduction).
- **SFS-EN 12457-3:** Two stage batch test at a liquid to solid ratio of 2 l/kg and 8 l/kg for materials with high solid content with particle size below 4 mm (without or with size reduction).
- **SFS-EN 12457-4:** One stage batch test at a liquid to solid ratio of 10 l/kg with particle size below 10 mm (without or with limited size reduction).

The following Table 7 summarizes the main conditions, parameters and ratios of these four standards. In this study is used only one of these standards, the SFS-EN 12457-3.

TABLE 7. The main conditions, parameters and ratios of the standard SFS-EN 12457/1-4

Standard	Sample weight (g)	Particle size (mm)	L/S (l/kg)	Contact time (h)
SFS-EN 12457-1	175	<4	2	24
SFS-EN 12457-2	90	<4	10	24
SFS-EN 12457-3	175	<4	2/8	6+18
SFS-EN 12457-4	90	<10	10	24

4.1.5 Standard SFS-EN 12457-3

According to the standard, this type of test is mainly used to determine and investigate the inorganic constituents of the waste without taking into account the particular characteristics of the non-polar organic constituents and the consequences of the microbiological processes in the degradable organic waste.

The process described in this test regulated by European Standards, will allow the production of eluates that will be analysed and characterised both chemically and physically.

Equipment, instruments and reagents

For the accomplishment of this test a series of equipment, instruments and reagents are needed (Table 8).

TABLE 8. Equipment, Instruments and reagents needed for the performance of standard SFS-EN 12457-3

Equipment	Instruments	Reagents
<ul style="list-style-type: none"> • Mixing device • Filtering device • Crushing equipment • Sieving equipment • A device for measuring conductivity • pH meter • Scale 	<ul style="list-style-type: none"> • Glass or plastic bottles (500ml, and 2000ml). • Filters • Test tubes • Beaker 	<ul style="list-style-type: none"> • Distilled water

Sampling and sample preparation

At least 2 kg of sample were collected in the sampling process, taking into account that the sample should be taken from different sides of the area to be as homogeneous and representative as possible.

This type of test shall be made from material that has a grain size of at least 95% of the whole mass, less than 4 mm, therefore the sample must be sieved. If the sample exceeds 5% of the oversized material, the entire oversized fraction has to be crushed. For that it can be used a mortar. In this process the sample may be too moist for crushing if this happens it is allowed to dry the sample. In this study this was not necessary, but was necessary to crush the sample until 4mm size of the particles was achieved.

Before starting the first leaching step, the dry matter content ratio, the moisture content ratio and the total mass of the sample (M_W) needed, were calculated following the next formulas:

$$DR = \frac{\text{measured sample amount after drying (g)} - \text{measured dish (g)}}{\text{measured sample amount before drying (g)}} \cdot 100\% \quad (1)$$

$$MC = 100 \cdot (M_W - M_D) / M_D \quad (2)$$

$$M_W = 100 \cdot M_D / DR \quad (3)$$

DR: Dry matter percent

MC: Moisture content ratio

M_D : The mass of the dried test portion (kg)

M_W : The mass of undried test portion (kg)

The results of these calculations are detailed in sections 5.1 and 5.2.

Procedure

First leaching step

To carry out the first leaching step is needed to place the mass of undried test portion calculated before in a bottle of 500 ml. Then according to the next formula (Formula 4), an amount of leachant solution (L_2) is added

with the aim of establish a liquid solid ratio $L/S=2 \text{ l/kg} \pm 2 \%$. To be able to establish this liquid - solid ratio, the bottle is agitated in a mixing device during $6 \text{ h} \pm 0,5 \text{ h}$.

$$L_2 = \left(2 - \frac{MC}{100}\right) \cdot M_D \quad (4)$$

After six hours, the capped bottle is let to stand with the objective to allow the suspended solids to settle for $15 \text{ min} \pm 5 \text{ min}$ (This time is the one that proposes the standard, but in the case of this study in both first and second leaching step 24h have been waited until the filtering process). These minutes or hours of stand make easier the subsequent filtering of the sample.

The eluates volume, conductivity ($\mu\text{S/cm}$) and pH are measured once the first liquid - solid separation step is done.

Second leaching step

A second bottle of 2 l of capacity is filled with the filters, the filtered parts of the first leaching step and an amount of leachant (L_8), to establish a cumulative liquid to solid ratio $L/S=10 \text{ l/kg} \pm 2 \%$ (Formula 5).

$$L_8 = 8 \times M_D \quad (5)$$

Again the bottle has to be mixed and let to stand for the following filter process, but in this step $18 \text{ h} \pm 0,5 \text{ h}$ are needed to establish this liquid - solid ratio.

As it has been made in the first leaching step the eluates volume, conductivity ($\mu\text{S}/\text{cm}$) and pH are measured with the objective of compare with the results with those obtained by testing laboratory.

4.2 Determination of Cr and Cu with Palintest photometer 7000se

Once the batch test is done, its possible to calculate the concentration of Chromium and Copper in each sample, with a photometer.

Palintest photometer 7000se is used to measure the colour intensity of the samples. Light from an incandescent lamp is passed through the test tube containing the sample solution, and then through a coloured filter onto a photocell. Light detected by the photocell is displayed as a digital response in form of concentration value.

When the chromium present in a sample is going to be analysed it must take into account that chromium may be present in hexavalent form as chromates and dichromates, or in trivalent form as chromium salts. In water supplies hexavalent chromium is a particularly objectionable constituent. Trivalent chromium, although relatively inert, is also regarded as undesirable.

In the case of copper, the test provides the opportunity to measure specifically the concentrations of free and chelated copper present in the sample solution.

The Palintest test provides a means of measuring chromium over the range 0-1mg/l and a means of measuring copper in natural and treated waters over the range 0-5 mg/l.

4.2.1 Method

Palintest chromicol method

In the Palintest chromicol method, hexavalent chromium salts in acidic conditions react with diphenylcarbazide to give a purpul coloured complex.

This provides a measure of the hexavalent chromium (Cr^{VI}) present in the sample. The reagents are provided in tablet form and the test is simply carried out by adding tablets to a sample of the water.

To determine total chromium (Cr^{III}) plus (Cr^{VI}) a fresh sample of the solution is oxidised using a powder reagent to convert the trivalent chromium to the hexavalent form. The test is then repeated to give a measure of the total soluble chromium content of the water. The difference between the two readings gives a measure of trivalent chromium concentrations and is measured using Palintest photometer.

Palintest coppercol method

In the Palintest Coppercol method copper salts are reduced to the cuprous form and then reacted with a 2,2 Biquinoline-4,4-dicarboxylic salt to form a purple coloured complex. This provides a measure of the free copper ions present in the sample. In the second stage of the test, a decomplexing agent is introduced and this induces a further reaction with any chelated copper compounds, which might be present.

The reagents are provided in tablet form and the test is simply carried out by adding tablets to a sample of the water. The intensity of colour produced in the test is proportional to the copper concentrations and is measured using a Palintest photometer.

4.2.2 Reagents and equipment

For each of two determinations some reagents and equipments are needed. Table 9 shows the information for chromium and table 10 for the copper.

TABLE 9. Reagents and equipment for the determination of chromium concentration

Hexavalent chromium	Trivalent and Total Chromium
<ul style="list-style-type: none"> • Palintest chromicol n° 1 Tablets • Palintest chromicol n° 2 Tablets • Palintest Photometer • Palintest Test Tubes, 10 mL glass (PT 515) 	<ul style="list-style-type: none"> • Palintest chromicol CR reagent (Spoon Pack) • Palintest pretreatment tube, 20 mL plastic (PT 508)

TABLE 10. Reagents and equipment for the determination of cooper concentration

Reagents	Equipment
<ul style="list-style-type: none"> • Palintest coppercol n° 1 Tablets • Palintest coppercol n° 2 Tablets 	<ul style="list-style-type: none"> • Palintest photometer • Round test tubes, 10 ml

4.2.3 Determination of hexavalent chromium

To determine the hexavalent chromium present in the 18 samples (9 from the first leaching step and the other 9 for the second leaching step) obtained in the batch test it's necessary to fill tubs of 10 ml with this solution and then add, mix and dissolve one chromicol n° 1 tablet and one chromicol n° 2 tablet. To allow full colour development the tube should stand for 10 min without disturbing and then is ready to be read in the photometer.

4.2.4 Determination of total chromium

To determine the total chromium in the samples the process is quite similar than the explained before. The differences are that in this case the tubes are of 20 ml and instead of add at first chromicol tablets now a spoon of chromicol CR powder is added. Once the tube is shaken and stands for two minutes the chromicol tablets are added. Subsequently, selecting a wavelength of 520 nm the concentration of total chromium is readed by the photometer.

4.2.5 Determination of copper

The test procedure for the determination of copper also is so similar to the previous two. A 10 ml tube is filled with the sample solution and the coppercol n° 1 tablet is added. Then, once the color or turbidity in the sample appears it is ready to read the free copper concentration. Then if the total copper value is required, only need to add the coppercol n° 2 tablet and read again the sample with the appropriate program.

4.3 Ecotoxicological test with Artemia Salina

4.3.1 Artemia Salina

Artemia salina is a kind of crustacean, which lives in salt water. This species it has probably not changed in 100 million years so it is considered one of the oldest species in the world. (Jiménez, Gelabert & Brito 2014.)

Artemia eggs remain inactive until they find the necessary environmental conditions for their growth. Once these eggs find the proper conditions it takes at least 24 hours before the first nauplii appears, but they do not reach adulthood until after 20-30 days. (Lagarto Parra, Silva Yhebra, Guerra Sardiñas & Iglesias Buela 2001)

Usually a very simple organism forms these species; they only have head, chest and abdomen. This simple organism and its primitive nervous system make them suitable for toxicological tests since the damage caused to the animal is small or practically non-existent. (Gilchrist 1960.) Even so, according to the Finnish law for test animal protection, *Artemia* can be used only for scientific or educational purposes. It is also important to make sure that is a minimum amount of animals are used and a minimum amount of pain, suffering or permanent damage are caused. (Animal Welfare Act 1430/2006)

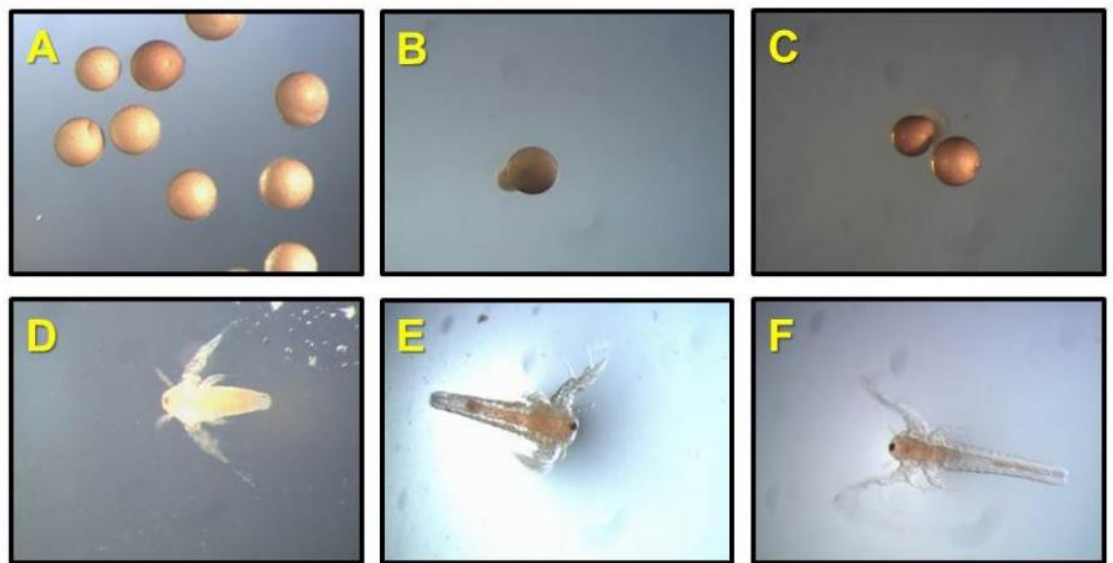


FIGURE 3. The process of growth of *Artemia Salina* (Lagarto Parra, Silva Yhebra, Guerra Sardiñas & Iglesias Buela 2001)

4.3.2 Reagents and equipment

To conduct this test, the reagents and equipment are shown in table below (Table 11).

TABLE 11. Reagents and equipment for the ecotoxicological test

Reagents	Equipment
<ul style="list-style-type: none"> • Solutions obtained in batch test • Salt solution 	<ul style="list-style-type: none"> • Cell plate • Petri dish • Pasteur pipette • Finn pipette

4.3.3 Procedure of ecotoxicological test

Before performing the test, the *Artemia Salina* has to be cultivated. In this study it has been used JBL Artemio pur eggs and JBL Artemio salt (a special culture solution for breed *artemia nauplii*, which contains microalgae as initial food and a buffer system to maintain the optimum pH level).

Three containers have been prepared with one liter of this salt solution and 5 spoonfuls of *Artemia pur* eggs. After three days the first *nauplii* begin to appear. Once enough *Artemias* have grown the test can start.

Each cell of the cell plate is filled with 10 *Artemias*, salt solution and leachant solution using a pipette (Table 12). At the moment that the cell plate is done, it is possible to record the first results. Every hour, the amount of *Artemias* that are still alive is observed with the objective of relating the mortality of the organism with the toxicity of the sample to be analysed.

The following table exemplifies the amounts of salt solution and leached solution required for the preparation of the analysis of samples number 1 (Power plant 1, bottom ash), number 2 (Power plant 2, gasifier bottom ash) and number 3 (Process heating plant, bottom ash) provided by Lahti Energy. This table is useful for both first and second leachant solutions.

TABLE 12. Amounts of leachant and salt solution

Solution obtained in batch test	The cell number of the cell plate	Amount of leachant solution (μl)	Amount of salt solution (μl)
1a, 1b, 1c	1	2500	500
	2	1500	1500
	3	500	2500
2a, 2b, 2c	4	2500	500
	5	1500	1500
	6	500	2500
3a, 3b, 3c	7	2500	500
	8	1500	1500
	9	500	2500
Control	10	0	3000

5 RESULTS AND DISCUSSION

5.1 Batch test results

Before doing the practical part of this thesis, as has been said before, some things like the amount of liquid that is needed or the dry weight should be calculated for the appropriate preparation of the samples and the test. Once this is done, the process can start.

5.1.1 Determination of dry weight

The dry weight of the samples is determined according to the SFS-EN 3008 standard, following a simple process. A petri dish is weighed and tared and then 10.0 g of soil sample is weighed. Later the petri dish is put into a heating chamber at 105 °C for 2 hours to remove any moisture in the sample. Having done that, the sample is allowed to cool down in an exsiccator for 2 hours more, and then the weight of the sample is measured to calculate the dry matter with the Formula 1 of section 4.1.2.2 of this thesis and the weights of Table 13.

TABLE 13. Dishes and samples weights

	Samples	DW	WS	WSA
1	Power plan 1, Bottom ash	0.8015 g	10.0005 g	8.4672 g
2	Power plan 2, Gasifier bottom ash	0.8016 g	10.0079 g	10.8062 g
3	Process heating plant, Bottom ash	0.7956 g	10.0062 g	10.7847 g

DW: Dish weight

WS: Weight sample amount

WSA: Weight sample amount after drying

Sample 1:

$$\text{Dry matter percent} = \frac{8,4672 \text{ g} - 0,8015 \text{ g}}{10,0005 \text{ g}} \cdot 100 \% = 76,65 \%$$

Sample 2:

$$\text{Dry matter percent} = \frac{10,8062 \text{ g} - 0,8016 \text{ g}}{10,0079 \text{ g}} \cdot 100 \% = 99,97 \%$$

Sample 3:

$$\text{Dry matter percent} = \frac{10,7847 \text{ g} - 0,7956 \text{ g}}{10,0062 \text{ g}} \cdot 100 \% = 99,83 \%$$

TABLE 14. Comparison of the dry matter percentage obtained by testing laboratory and the results obtained in the practical part of this thesis.

	Dry matter percentage testing laboratory	Dry matter percentage
Power plant 1, bottom ash	69,2%	76,65%
Power plant 2, gasifier bottom ash	100,0%	99,97%
Process heating plant, bottom ash	99,6%	99,83%

These results are very similar to the obtained by the testing laboratory, as it could be seen in Table 14 the sample with the highest percentage of dry matter is the sample 2 and in the other hand the sample with the lower percentage is the number 1.

5.1.2 Preparation of test portion

Following Formulas 2, 3, 4 and 5 is possible to calculate the portion of liquid necessary for the first leaching step and the second leaching step.

Sample1:

$$M_W = 100 \% \cdot \frac{0,175 \text{ kg}}{76,65 \%} = 0,2283 \text{ kg}$$

$$MC = 100 \% \cdot \frac{0,2283 \text{ kg} - 0,175 \text{ kg}}{0,175 \text{ kg}} = 30,4571 \%$$

$$L_2 = \left(2 - \frac{30,4571 \%}{100 \%}\right) \cdot 0,175 \text{ kg} = 0,2967 \text{ kg} = 0,2967 \text{ L}$$

$$L_8 = 8 \times 0,175 \text{ kg} = 1,4 \text{ kg} = 1,4 \text{ L}$$

Sample2:

$$M_W = 100 \% \cdot \frac{0,175 \text{ kg}}{99,96 \%} = 0,1751 \text{ kg}$$

$$MC = 100 \% \cdot \frac{0,1751 \text{ kg} - 0,175 \text{ kg}}{0,175 \text{ kg}} = 0,0571 \%$$

$$L_2 = \left(2 - \frac{0,0571 \%}{100 \%}\right) \cdot 0,175 \text{ kg} = 0,3499 \text{ kg} = 0,3499 \text{ L}$$

$$L_8 = 8 \times 0,175 \text{ kg} = 1,4 \text{ kg} = 1,4 \text{ L}$$

Sample3:

$$M_W = 100 \% \cdot \frac{0,175 \text{ kg}}{99,82 \%} = 0,1753 \text{ kg}$$

$$MC = 100 \% \cdot \frac{0,1753 \text{ kg} - 0,175 \text{ kg}}{0,175 \text{ kg}} = 0,1714 \%$$

$$L_2 = \left(2 - \frac{0,1714 \%}{100 \%} \right) \cdot 0,175 \text{ kg} = 0,3497 \text{ kg} = 0,3497 \text{ L}$$

$$L_8 = 8 \times 0,175 \text{ kg} = 1,4 \text{ kg} = 1,4 \text{ L}$$

5.1.3 Conductivity and pH results

Conductivity and pH were analysed from each sample in the first and the second leaching step and before doing any treatment to the samples. The results of these three situations are shown in Figures 4, 5 and 6.

The results of the three samples show that the pH values in each of them were alkaline. However sample number two has a higher pH than the other two. According to Van Herck and Vandecasteele (2001), an alkaline pH indicates that part of the dissolved metals occur as basic metal salts, oxides, and even as hydroxides and/or carbonates. (Manskinen 2013.)

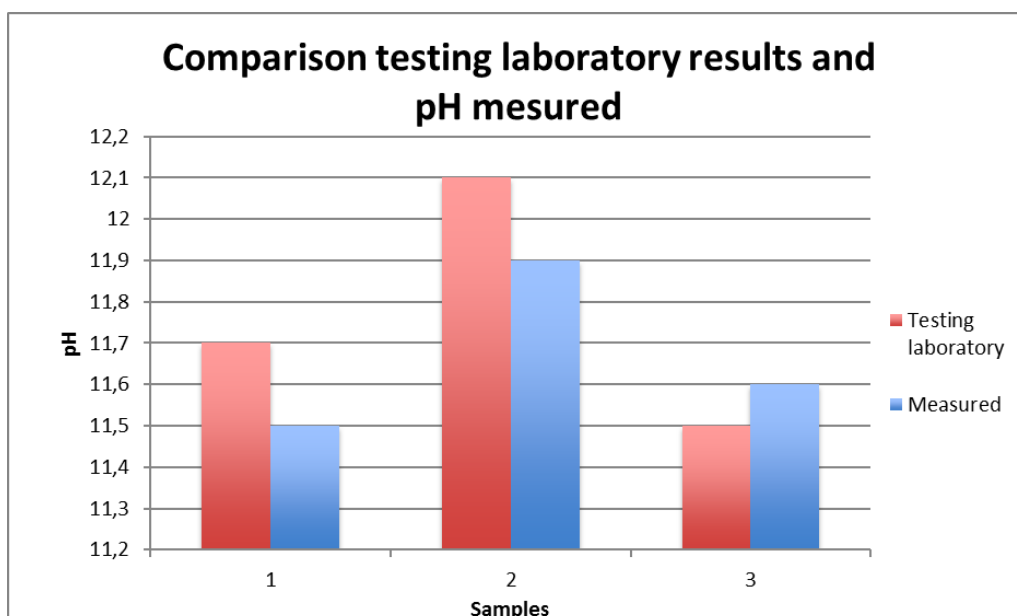


FIGURE 4. Comparison of the pH results obtained in the practical part of this study and by the testing laboratory

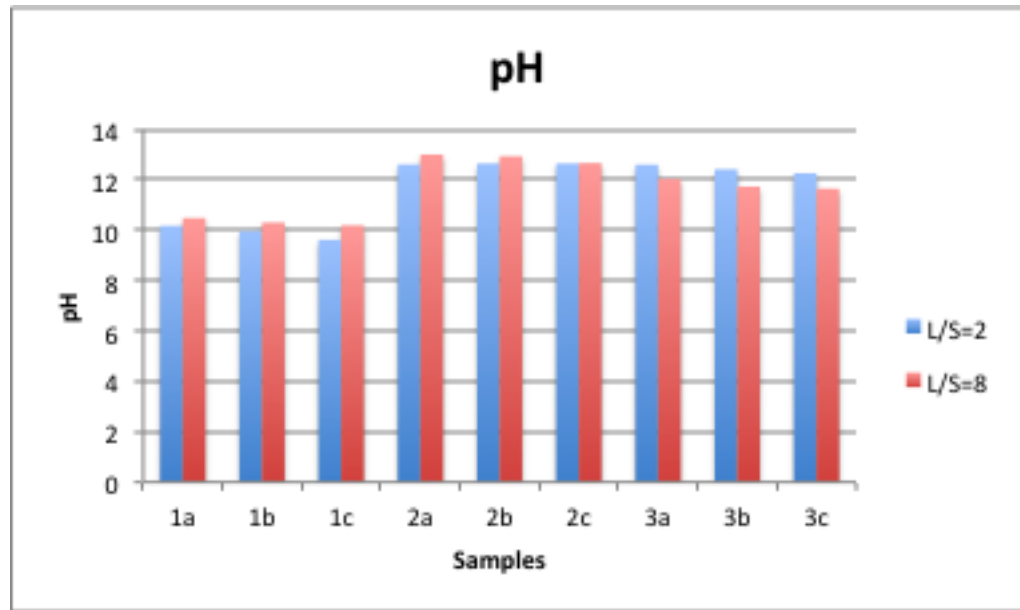


FIGURE 5. Representation of pH results in the first and second leaching step

Conductivity is defined as the ability of a substance to conduct electrical current and the most common measurement unit is microSiemens/centimeters ($\mu\text{S}/\text{cm}$). This value is directly proportional on the amount of concentration of dissolved substance (TDS). So it could be seen in Figure 6 and Appendix 1, the sample 2 which is the one with the highest TDS value (19100 mg/kg) is in turn the sample with the highest conductivity in both first and second leaching steps.

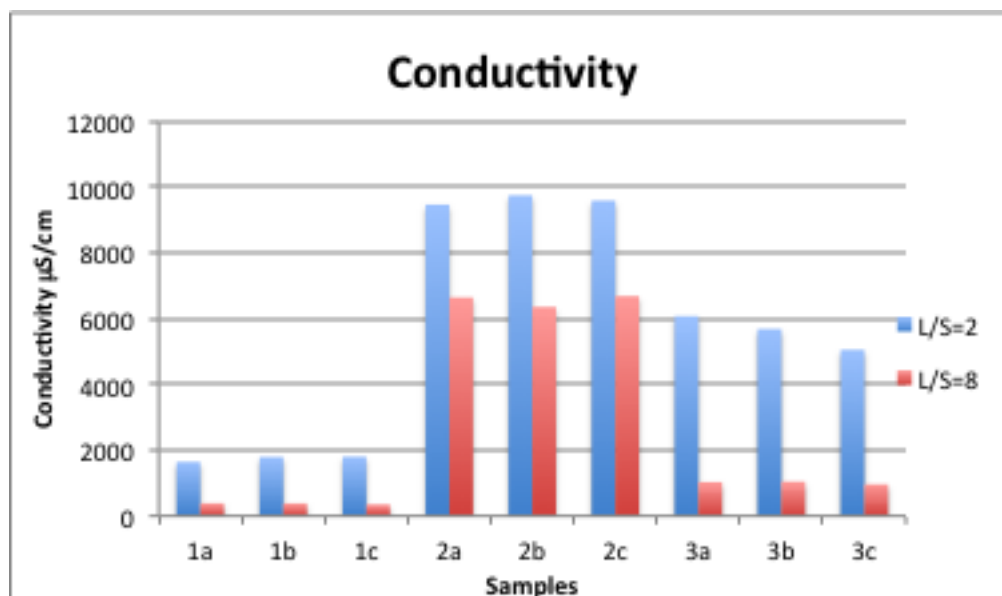


FIGURE 6. Representation of conductivity results in the first and second leaching step

5.2 Photometer results

As has already been said, chromium may be present in the samples in hexavalent form as chromates and dichromates, or in trivalent form as chromium salts. In this case of the study as is shown in figures 7 and 8, both forms are present in the samples.

One more time the sample number 2 is the one with the highest concentration of chromium. It will be seen later whether or not this fact affects the results of the ecotoxicological test. By the time, if the results are compared with the limit values of the Government Decree (591/2006), the samples 2 and 3 of the first leaching step and the sample 2 of the second leaching step exceed the limit value for Leaching (L/S=10 l/kg) Covered structure (0,5 mg/kg).

All values of the concentrations, both those of the chromium concentrations and those of the copper concentrations, are detailed in Appendix 3. Also in Appendix 4 it could be seen the representations of hexavalent, trivalent and total chromium separate.

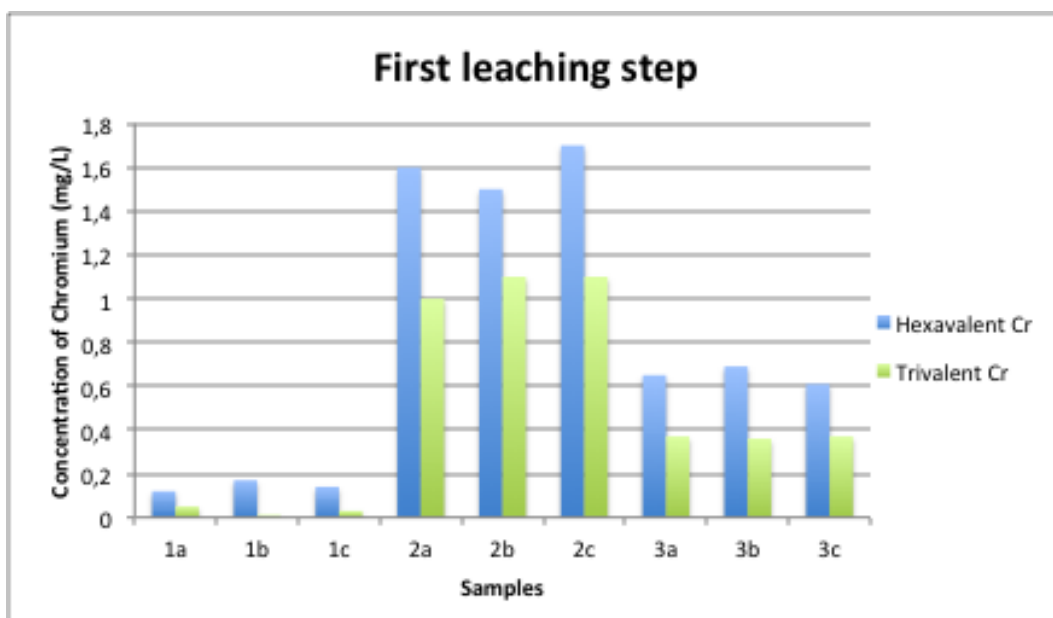


FIGURE 7. Concentration of chromium in the first leaching step

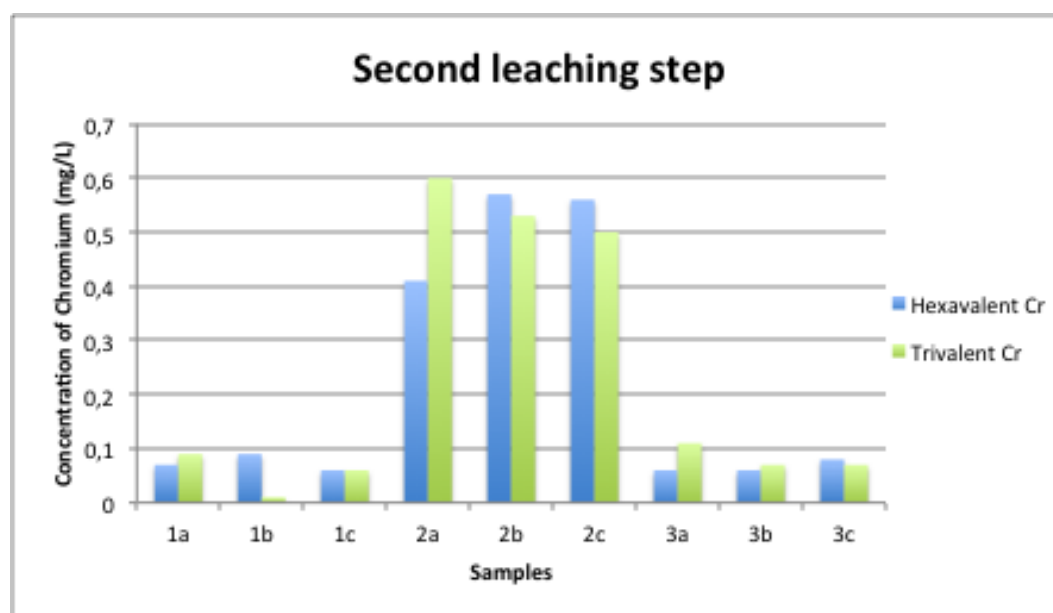


FIGURE 8. Concentration of chromium in the second leaching step

In the figure 9 is represented the concentration of copper for each one of the samples of this thesis. The copper concentrations in the first step of the batch test vary considerably in the second step. There is practically a difference of half a unit.

Power plant 2 gasifier bottom ash sample, again have the highest concentration of copper and also exceed the limits set by the Government Decree (591/2006). And in this case, not only overtake the limits for covered structure also surpass the limit of Leaching (L/S=10 l/kg) Paved structure (6 mg/kg). If this data want to be checked in an easier form as it has been said before, all the detailed values are in the Appendix 3.

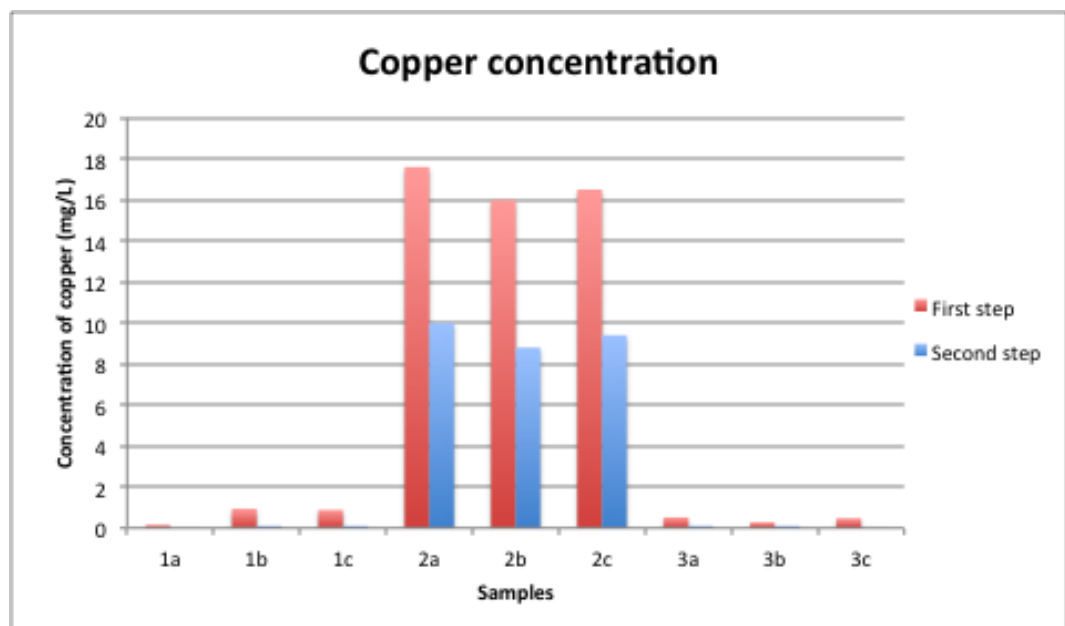


FIGURE 9. Concentration of copper in the first and second leaching step

5.3 Ecotoxicological test results

One of the main objectives of this test is to demonstrate whether *Artemia Salina* is a good option or not, to determine the possible utilization of ash and concrete waste in geotechnical construction. For this, six tests have been carried out at different concentrations of the pollutant solution and in different stages of the life of *Artemia Salina*.

First of all, the samples obtained in the first leaching step have been analysed and then the same process has been done with the samples of the second leaching step.

To check how the age of *Artemia Salina* affects the test, the first day samples 1a, 2a and 3a were analysed, second day samples 1b, 2b and 3b, and finally the third day samples 1c, 2c and 3c were analysed. The results that have been obtained of all this tests, are shown below in the form of different graphs (Figures 10 and 11).

As can be seen in Figure 10 (showing the amount of live *Artemia Salina* as a function of time) at a higher age of the organism, there is less resistance to toxics. On the first day, *Artemia Salina* lasted approximately 7 hours before dying, while on their third day of life, following the same conditions as in the first one, they lasted 4 hours. Also, it has been observed that the sample with the highest toxicity is number 2.

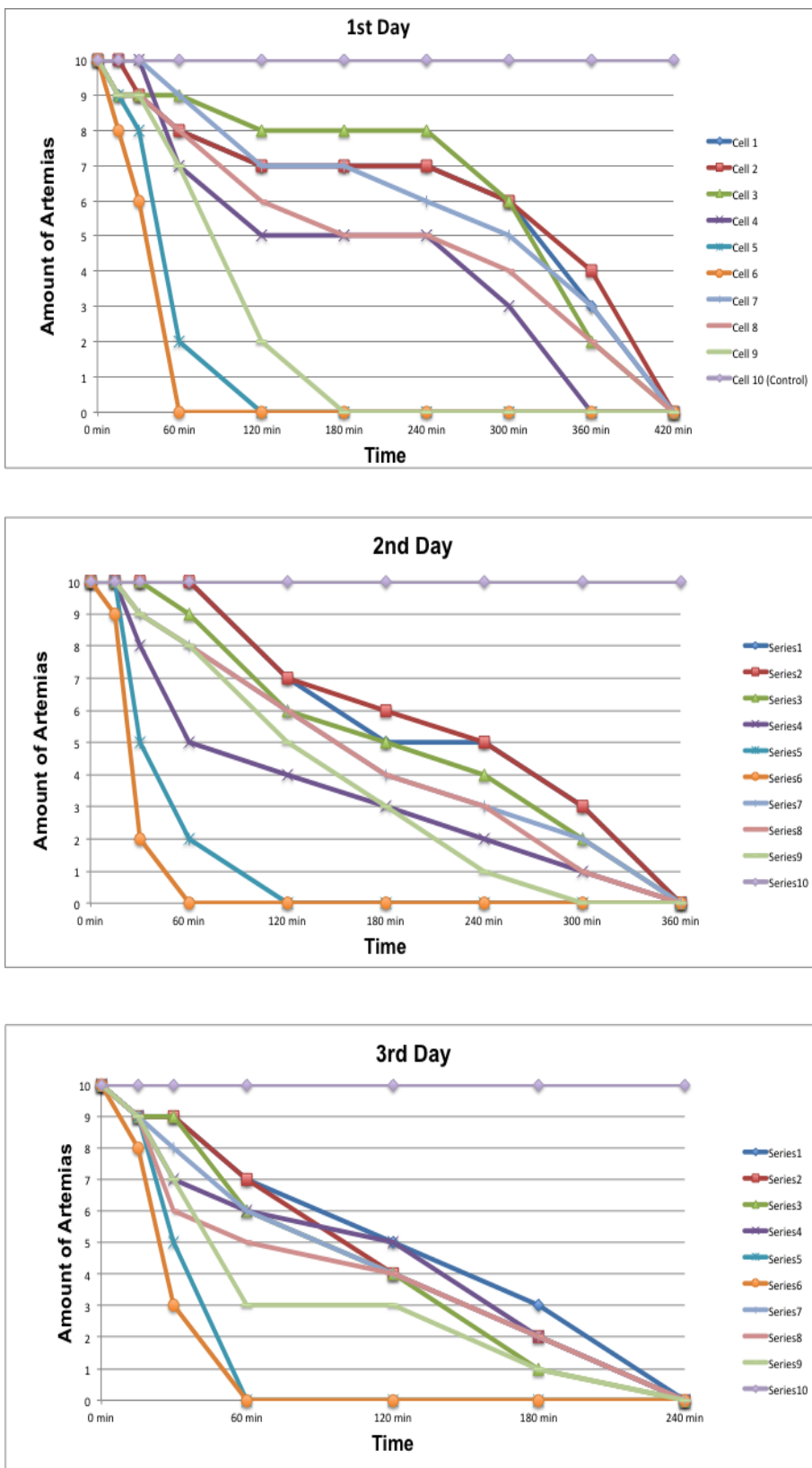


FIGURE 10. Representation of the amount of live Artemia Salina as a function of time in 3 different days (testing the first leachant solution)

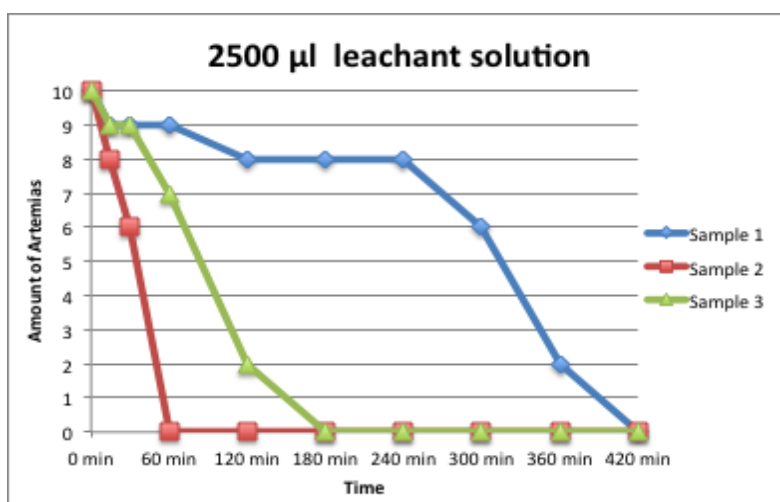
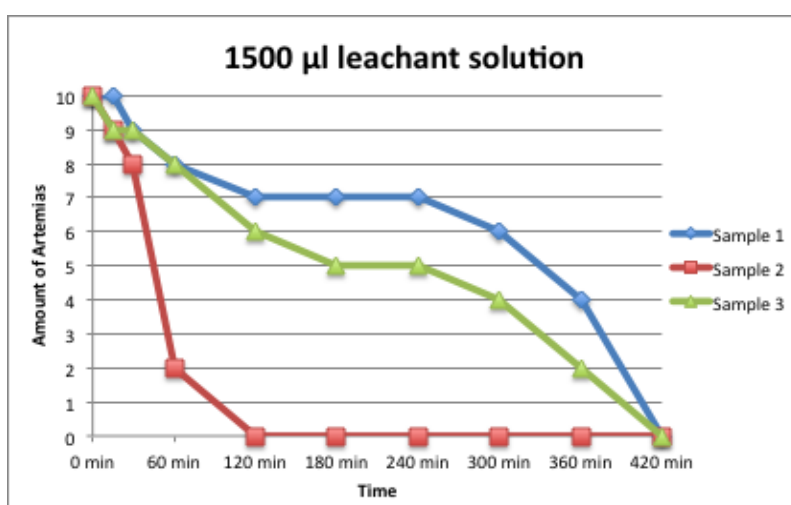
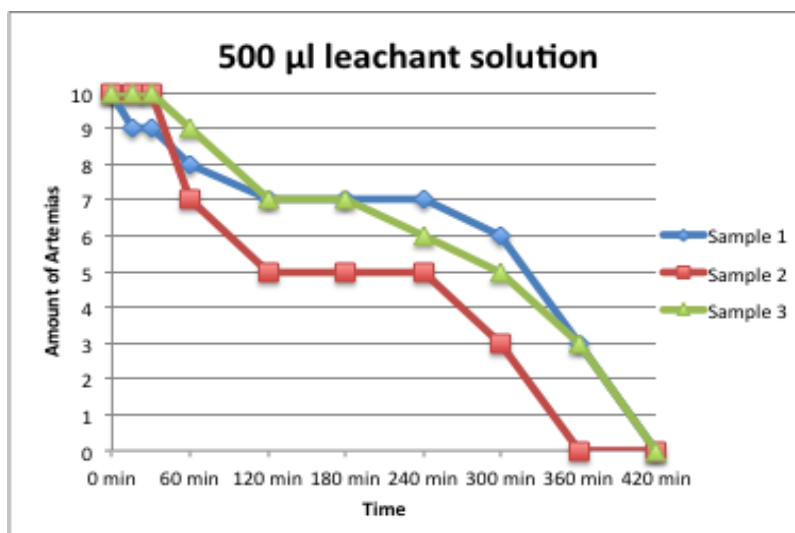


FIGURE 11. Representation of the mortality of the Artemia Salina for each one of the concentrations of leachant solution the first day of testing (testing the first leachant solution)

In Figure 11, which represents the mortality of *Artemia Salina* for each of the concentrations of leachant solution, it can be seen that in all three graphs, the red line (sample 2) always goes below the other two, indicating that because of a higher toxicity of the sample, the invertebrate organism dies in a shorter time. One of the reasons of this high toxicity could be to copper present in the sample.

During the process, it was observed that cells number 4, 5 and 6 presented a kind of turbidity, possibly on account of the reaction of the copper with the salt of the water. This turbidity wasn't present in the other samples, which is an indicative that the concentration of copper in sample number 2 is higher than in the other two, as well as the concentration of chromium which is also higher in this sample. This fact can be observed in Figures 7, 8 and 9 where the concentration of copper and chromium in sample 2 is much higher than in the other two samples.

The reason for all these deaths is that these aquatic organisms accumulate copper, chromium and other harmful substances by diffusion. These substances penetrate through the cell membrane (a very fine skin, which makes them especially sensitive to toxics) of *Artemia Salina* following the Fick's laws of diffusion (diffusion is the movement of a substance from a region of high concentration to a region of low concentration) and causing damage and different alterations in these organisms, ranging from difficulties in mobility to death.

On the other hand, the results obtained from the second part of the ecotoxicological test (toxicological analysis of the leachant solution of the second step of the batch test) do not differ significantly from those obtained in the first part.

The high concentration of chromium and copper of power plant 2 also affect the *Artemias*, causing difficulties in the mobility and the death of the organisms. However, in this second test, the concentration of the harmful substances is lower than in the first case so the life of the tested animals is longer than in the first ecotoxicological test. If in the first test the *artemia*

lasted approximately between 7 and 4 hours depending on the age of the organism, in this second test they can live between a 24 and 12 hours before dying.

Another difference is that in this last test there is no difference between the second and third day of the life of Artemia. In both cases, it took 12 hours until the last artemia died. Likewise, it is confirmed that as was expected, at higher concentration of leaching solution there is higher mortality. All this can be seen in graphic form in Figures 12 and 13. Also if more information is needed it is possible to see the Appendix 5, that contain all the results of the ecotoxicological test.

Morover, to know exactly wich is the concentration of harmful substances analysed with the photometer in each of the cells of ecotoxicological test, it has been used Formula 11.

$$c_i \cdot V_i = c_f \cdot V_f \quad (11)$$

C_i: Initial concentration

C_f: Final concentration

V_i: Initial volume

V_f: Final volume

Example of the first cell:

$$0,17 \frac{mg}{L} \cdot 0,0005L = C_f \cdot 0,003 L$$

$$C_f = \frac{0,17 \frac{mg}{L} \cdot 0,0005L}{0,003 L} = 0,0283 mg/L$$

As it could be seen in Tables 15 and 16, the Artemias Salina died so quickly in the cells 4, 5 and 6, because these cells always have the higher concentrations of copper and chromium. For example the concentration of

copper in the previous cells reaches values of up to 14 mg/l, whereas in cells 1, 2 and 3 it does not exceed 1mg/l.

TABLE 15. Concentration of chromium in each cell

First leaching step (L/S=2). Concentration of Chromium				Second leaching step (L/S=8). Concentration of Chromium			
Samples	Amount of leachant solution (μ l)			Samples	Amount of leachant solution (μ l)		
	500	1500	2500		500	1500	2500
1a	0.03 mg/l	0.09 mg/l	0.14 mg/l	1a	0.03 mg/l	0.08 mg/l	0.13 mg/l
1b	0.03 mg/l	0.09 mg/l	0.15 mg/l	1b	0.02 mg/l	0.05 mg/l	0.08 mg/l
1c	0.03 mg/l	0.09 mg/l	0.14 mg/l	1c	0.02 mg/l	0.06 mg/l	0.10 mg/l
2a	0.43 mg/l	1.30 mg/l	2.17 mg/l	2a	0.17 mg/l	0.51 mg/l	0.84 mg/l
2b	0.43 mg/l	1.30 mg/l	2.17 mg/l	2b	0.18 mg/l	0.55 mg/l	0.92 mg/l
2c	0.47 mg/l	1.40 mg/l	2.33 mg/l	2c	0.18 mg/l	0.53 mg/l	0.88 mg/l
3a	0.17 mg/l	0.51 mg/l	0.85 mg/l	3a	0.03 mg/l	0.09 mg/l	0.14 mg/l
3b	0.18 mg/l	0.53 mg/l	0.88 mg/l	3b	0.02 mg/l	0.07 mg/l	0.11 mg/l
3c	0.16 mg/l	0.49 mg/l	0.82 mg/l	3c	0.03 mg/l	0.08 mg/l	0.13 mg/l

TABLE 16. Concentration of copper in each cell

First leaching step (L/S=2). Concentration of Copper				Second leaching step (L/S=8). Concentration of Copper			
Samples	Amount of leachant solution (μ l)			Samples	Amount of leachant solution (μ l)		
	500	1500	2500		500	1500	2500
1a	0.03 mg/l	0.08 mg/l	0.13 mg/l	1a	0.00 mg/l	0.00 mg/l	0.00 mg/l
1b	0.16 mg/l	0.47 mg/l	0.78 mg/l	1b	0.00 mg/l	0.01 mg/l	0.02 mg/l
1c	0.15 mg/l	0.45 mg/l	0.74 mg/l	1c	0.00 mg/l	0.01 mg/l	0.02 mg/l
2a	2.93 mg/l	8.80 mg/l	14.67 mg/l	2a	1.67 mg/l	5.00 mg/l	8.33 mg/l
2b	2.67 mg/l	8.00 mg/l	13.33 mg/l	2b	1.47 mg/l	4.40 mg/l	7.33 mg/l
2c	2.75 mg/l	8.25 mg/l	13.75 mg/l	2c	1.57 mg/l	4.70 mg/l	7.83 mg/l
3a	0.09 mg/l	0.26 mg/l	0.43 mg/l	3a	0.00 mg/l	0.01 mg/l	0.02 mg/l
3b	0.05 mg/l	0.14 mg/l	0.23 mg/l	3b	0.01 mg/l	0.02 mg/l	0.03 mg/l
3c	0,08 mg/l	0.24 mg/l	0.40 mg/l	3c	0.00 mg/l	0.00 mg/l	0.00 mg/l

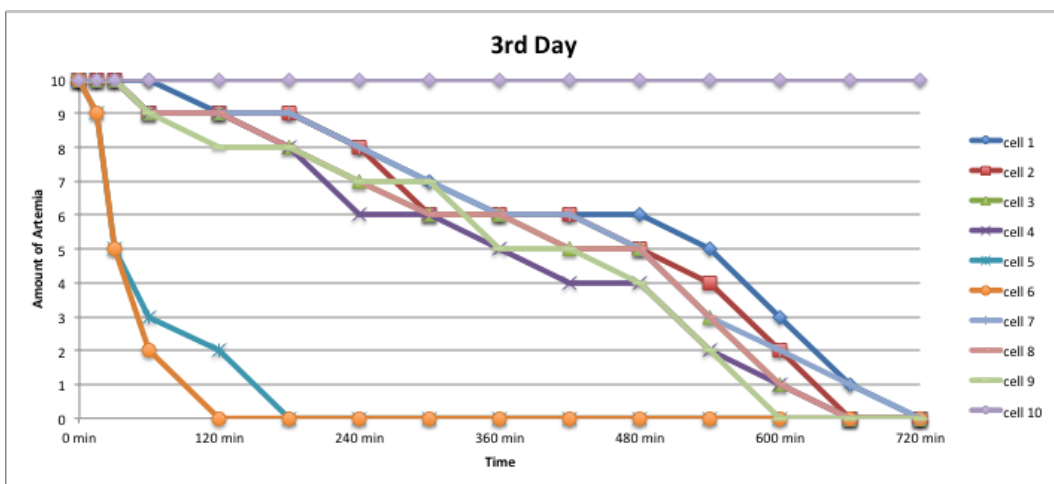
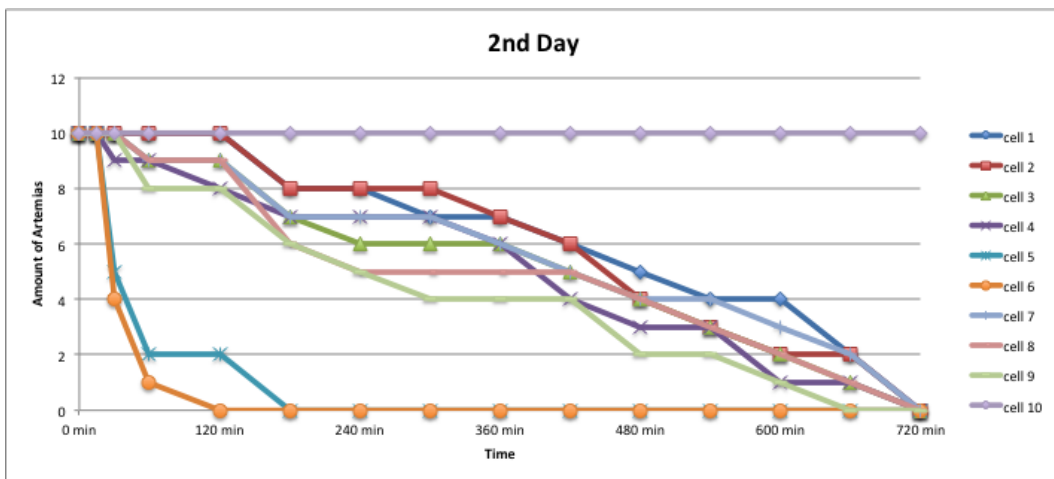
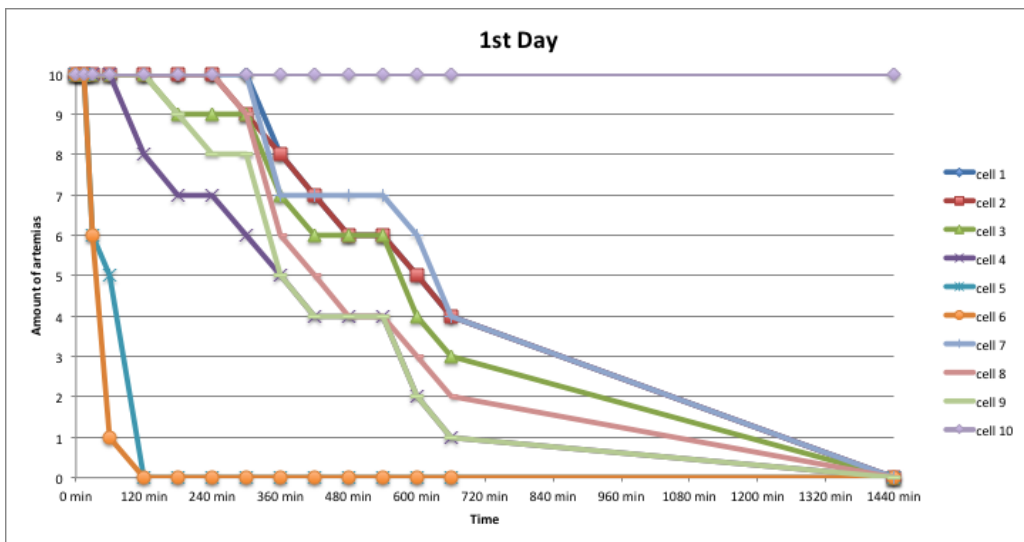


FIGURE 12. Representation of the amount of live Artemia Salina as a function of time in 3 different days (testing the second leachant solution)

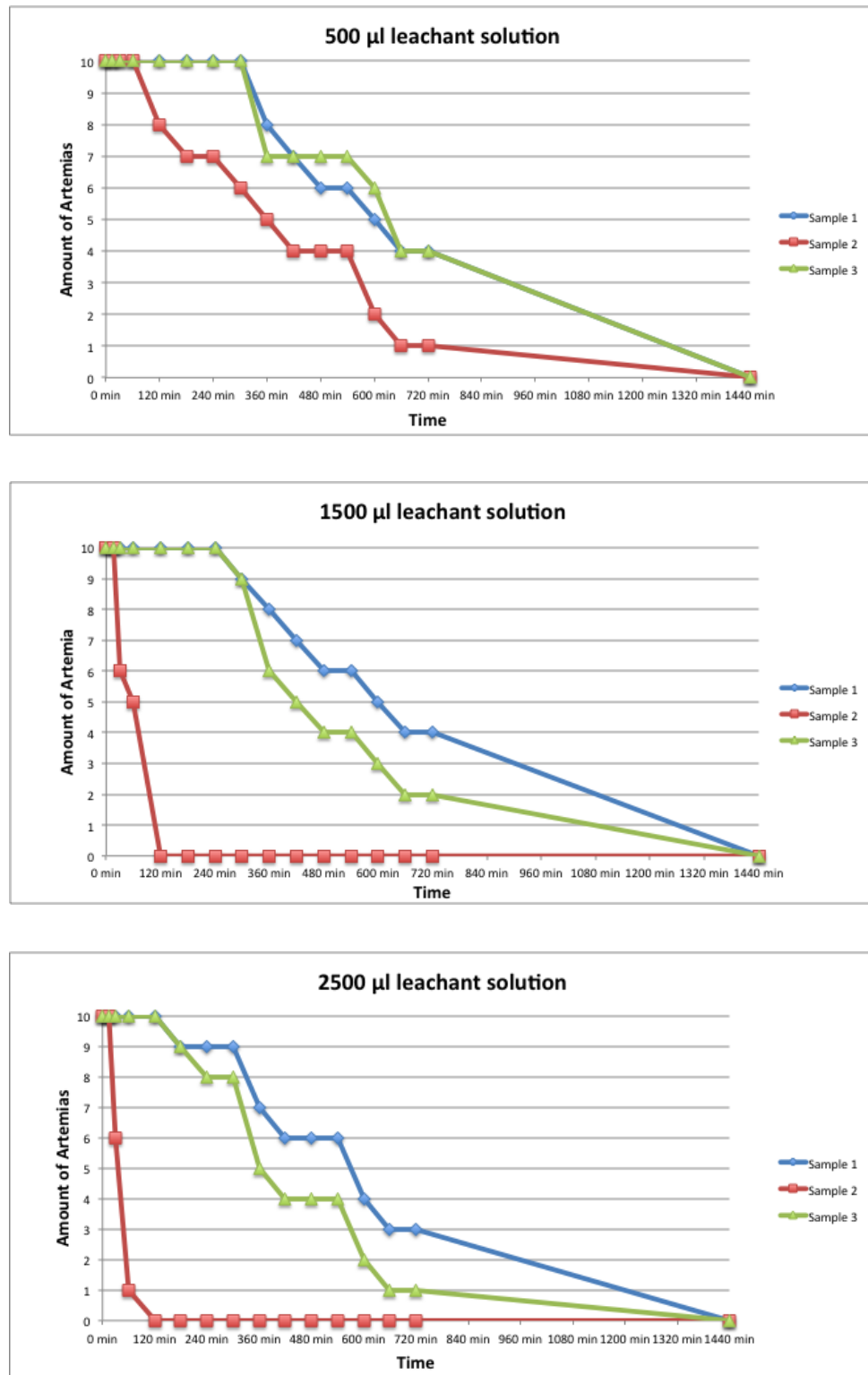


FIGURE 13. Representation of the mortality of the Artemia Salina for each one of the concentrations of leachant solution the first day of testing (testing the second leachant solution)

6 CONCLUSIONS

This study has provided a general view of the field of the utilization of concrete and ash waste in geotechnical construction. Information about the legislation, methods, standards and treatments in Finland and Spain has been the basis for the realization of this thesis.

Although it has been seen that both countries must increase their recycling rates and improve their reuse processes with the aim of reducing the environmental pollution, the comparison between these two countries has shown that although the two countries belong to the EU and therefore should be governed the same or at least similar laws, they differ in many aspects. While Finland has stricter regulations and its recycling rate is between 40-45%, Spain with 5% is in the last positions in the EU along with Greece and Portugal. Spain is a country that still has a long way to go if it wants to reduce its percentage of pollution on the contrary, Finland is near to the best countries (Belgium, Netherland and Denmark) in this field.

In the analyses that have been made, it is observed that all the samples tested present some type of harmful substance. However, only sample number 2 exceeds the limits established by the Government Decree (591/2006). In this moment reuse need an environmental permit, but because of low solubility the reuse is possible and also has been realized. These results obtained in the practical part coincide with those which have gotten by the testing laboratory. With respect to the other two samples, can be reused and therefore converted into by-products or raw materials provided that the laws, standards and regulations explained in this thesis are followed.

The ecotoxicological test with *Artemia Salina* confirms that sample number 2 is the most toxic. At the same time the test demonstrates that this kind of test is a good method of analysis. The tests also provided evidence that variables such as the sample concentration or the age of the organism tested are directly proportional to the mortality of *Artemia Salina*. That is,

the higher concentration of the sample or the older the organism is, the higher the rate of mortality is.

The fact of calculating the concentration in each of the cells has provided a better view of how polluting sample number 2 can become. As well as has given an idea of the maximum concentration that *Artemia Salina* can withstand before dying. For example looking Tables 15 and 16 it can be seen that in case of copper at a concentration of 2.93 mg/l more than a half of the *Artemias Salina* are dead in the time of one hour. On the other hand, in case of chromium the maximum concentration that *Artemias* can withstand before half of them are dead in one hour is 0,43 mg/l.

Even so, to complement the results of this thesis, it is proposed to carry out an alternative study. If the analysis time would be fixed and only the concentrations of the sample to be analyzed were varied it would be possible to perform a regression line of the percentage of dead *Artemia Salina* at the end of the test for each of the concentrations. This regression line would allow calculating the LC50 and therefore to estimate to which concentration the death of 50% of *Artemias Salina* of the study takes place.

Thanks to the use of the photometer, it has been verified that this toxicity and mortality are due in large part to the presence of chromium and copper in the samples. Copper and chromium are responsible for the death of *Artemias Salina* since these, following the diffusion laws, incorporate such harmful substances into their organism.

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APPENDICES

APPENDIX 1 Tables of testing laboratory results

APPENDIX 2 Tables of the pH and conductivity results obtained in the laboratory.

APPENDIX 3 Tables of the chromium and copper concentrations results

APPENDIX 4 Representations of chromium and copper concentrations

APPENDIX 5 Table ecotoxicological test results

APPENDIX 1

Power plant 1, bottom ash				
Two stage batch leaching test			Soluble concentration after batch test	
pH		Conductivity		<ul style="list-style-type: none"> • DOC: 28 mg/kg dm • TDS: 3840 mg/kg dm • Chlorides (Cl): 21 mg/kg dm • Sulphate (SO₄): 250 mg/kg dm • Fluoride (F): <5.0 mg/kg dm • Phenol index: <0.1 mg/kg dm • Arsenic (As): <0.01 mg/kg dm • Lead (Pb): <0.01 mg/kg dm • Cadmium (Cd): <0.003 mg/kg ka • Chromium (Cr): 0.20 mg/kg dm • Copper (Cu): <0.05 mg/kg dm • Nickel (Ni): <0.01 mg/kg dm • Mercury (Hg): <0.002 mg/kg dm • Zinc (Zn): <0.1 mg/kg dm • Antimony (Sb): <0.01 mg/kg dm • Barium (Ba): 14 mg/kg dm • Molybdenum (Mo): 0.26 mg/kg dm • Selenium (Se): 0.02 mg/kg dm • Vanadium (V): <0.1 mg/kg dm
L/S=2	L/S=8	L/S=2	L/S=8	
12	11,5	4000 µS/cm	1200 µS/cm	

Power plant 2, gasifier bottom ash				
Two stage batch leaching test			Soluble concentration after batch test	
pH		Conductivity		
L/S=2	L/S=8	L/S=2	L/S=8	
12,6	12,4	9960 μ S/cm	8650 μ S/cm	<ul style="list-style-type: none"> • DOC: 63 mg/kg dm • TDS: 19100 mg/kg dm • Chlorides (Cl): 500 mg/kg dm • Sulphate (SO₄): 47 mg/kg dm • Fluoride (F): <5.0 mg/kg dm • Phenol index: <0.1 mg/kg dm • Arsenic (As): <0.01 mg/kg dm • Lead (Pb): 1.3 mg/kg dm • Cadmium (Cd): <0.003 mg/kg dm • Chromium (Cr): 0.03 mg/kg dm • Copper (Cu): 6.2 mg/kg dm • Nickel (Ni): 0.02 mg/kg dm • Mercury (Hg): <0.002 mg/kg dm • Zinc (Zn): 2.5 mg/kg dm • Antimony (Sb): 0.16 mg/kg dm • Barium (Ba): 53 mg/kg dm • Molybdenum (Mo): 0.10 mg/kg dm • Selenium (Se): 0.02 mg/kg dm • Vanadium (V): <0.1 mg/kg dm

Process heating plant, bottom ash				Soluble concentration after batch test
Two stage batch leaching test			pH	
Conductivity		L/S=2		
L/S=2	L/S=8		L/S=2	L/S=8
11,7	11,2	2650 μ S/cm	760 μ S/cm	<ul style="list-style-type: none"> • DOC: 8,6 mg/kg dm • TDS: 3560 mg/kg dm • Chlorides (Cl): 15 mg/kg dm • Sulphate (SO₄): 280 mg/kg dm • Fluoride (F): <5.0 mg/kg dm • Phenol index: <0.1 mg/kg dm • Arsenic (As): <0.01 mg/kg dm • Lead (Pb): <0.01 mg/kg dm • Cadmium (Cd): <0.003 mg/kg dm • Chromium (Cr): 0.17 mg/kg dm • Copper (Cu): <0.05 mg/kg dm • Nickel (Ni): <0.01 mg/kg dm • Mercury (Hg): <0.002 mg/kg dm • Zinc (Zn): <0.1 mg/kg dm • Antimony (Sb): <0.01 mg/kg dm • Barium (Ba): 1.3 mg/kg dm • Molybdenum (Mo): 0.14 mg/kg dm • Selenium (Se): <0.01 mg/kg dm • Vanadium (V): 0.12 mg/kg dm

APPENDIX 2

Sample1: Power plant 1, bottom ash					
		pH		Conductivity	
		L/S=2	L/S=8	L/S=2	L/S=8
1a		10,175	10,483	1645 $\mu\text{S/cm}$	388 $\mu\text{S/cm}$
1b		9,968	10,305	1793 $\mu\text{S/cm}$	386 $\mu\text{S/cm}$
1c		9,620	10,195	1801 $\mu\text{S/cm}$	355 $\mu\text{S/cm}$
Sample 2: Power plant 2, gasifier bottom ash					
		pH		Conductivity	
		L/S=2	L/S=8	L/S=2	L/S=8
2a		12,610	13,005	9460 $\mu\text{S/cm}$	6640 $\mu\text{S/cm}$
2b		12,648	12,936	9740 $\mu\text{S/cm}$	6350 $\mu\text{S/cm}$
2c		12,649	12,667	9580 $\mu\text{S/cm}$	6680 $\mu\text{S/cm}$
Sample 3: Process heating plant, bottom ash					
		pH		Conductivity	
		L/S=2	L/S=8	L/S=2	L/S=8
3a		12,602	12,037	6070 $\mu\text{S/cm}$	1030 $\mu\text{S/cm}$
3b		12,420	11,732	5680 $\mu\text{S/cm}$	1044 $\mu\text{S/cm}$
3c		12,262	11,649	5050 $\mu\text{S/cm}$	964 $\mu\text{S/cm}$

APPENDIX 3

Concentration of chromium L/S=2				
Samples		Hexavalent chromium	Trivalent chromium	Total chromium
Sample 1: Power plant 1, bottom ash	1a	0,12 mg/L	0,05 mg/L	0,17 mg/L
	1b	0,17 mg/L	0,01 mg/L	0,18 mg/L
	1c	0,14 mg/L	0,03 mg/L	0,17 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	1,6 mg/L	1 mg/L	2,6 mg/L
	2b	1,5 mg/L	1,1 mg/L	2,6 mg/L
	2c	1,7 mg/L	1,1 mg/L	2,8 mg/L
Sample 3: Process heating plant, bottom ash	3 ^a	0,65 mg/L	0,37 mg/L	1,02 mg/L
	3b	0,69 mg/L	0,36 mg/L	1,05 mg/L
	3c	0,61 mg/L	0,37 mg/L	0,98 mg/L

Concentration of chromium L/S=8				
Samples		Hexavalent chromium	Trivalent chromium	Total chromium
Sample 1: Power plant 1, bottom ash	1a	0,07 mg/L	0,09 mg/L	0,16 mg/L
	1b	0,09 mg/L	0,01 mg/L	0,10 mg/L
	1c	0,06 mg/L	0,06 mg/L	0,12 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	0,41 mg/L	0,6 mg/L	1,01 mg/L
	2b	0,57 mg/L	0,53 mg/L	1,1 mg/L
	2c	0,56 mg/L	0,5 mg/L	1,06 mg/L
Sample 3: Process heating plant, bottom ash	3a	0,06 mg/L	0,11 mg/L	0,17 mg/L
	3b	0,06 mg/L	0,07 mg/L	0,13 mg/L
	3c	0,08 mg/L	0,07 mg/L	0,15 mg/L

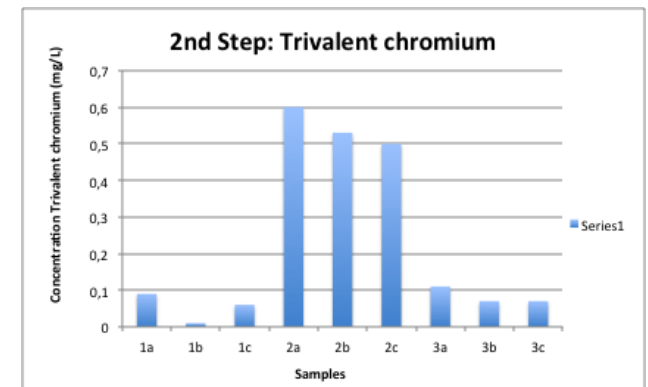
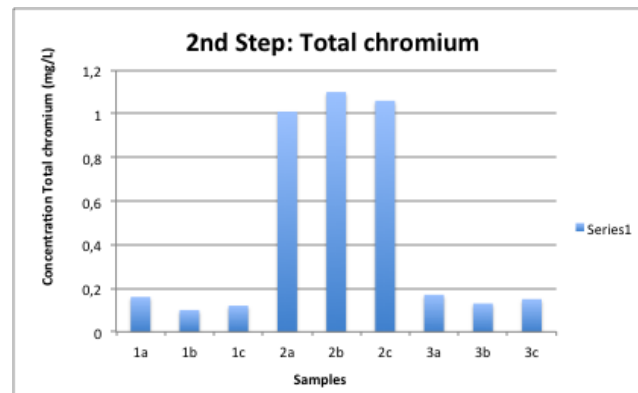
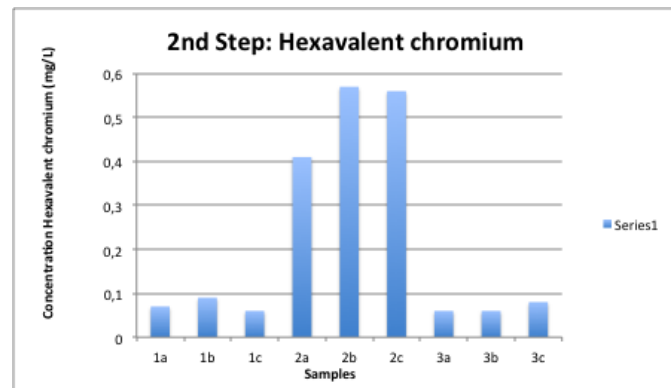
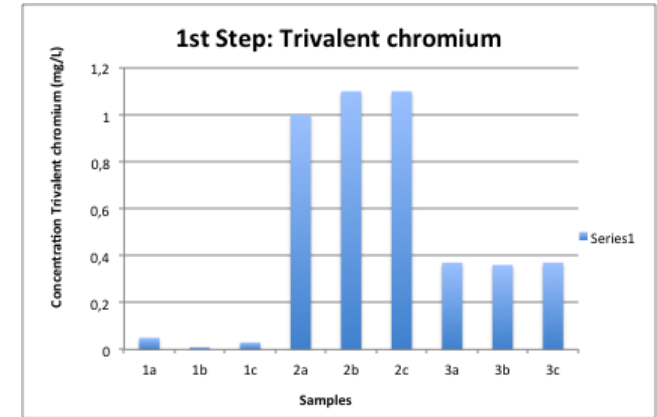
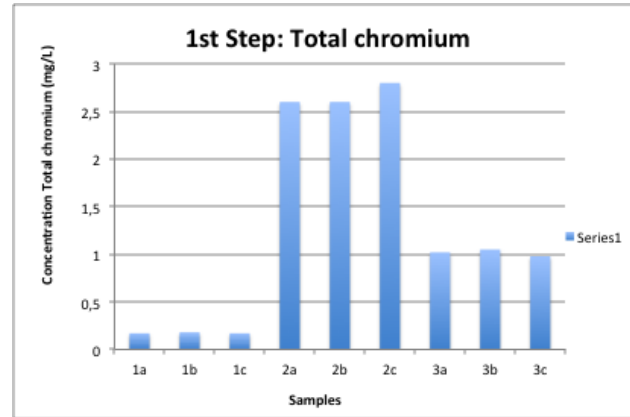
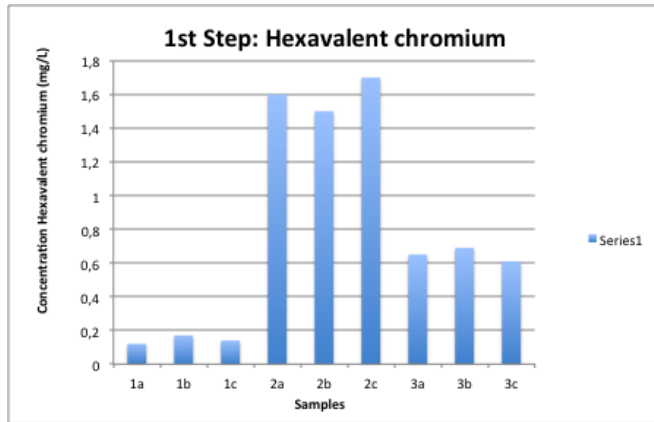
Concentration of Copper Free L/S=2		
Sample1: Power plant 1, bottom ash	1a	0,10 mg/L
	1b	0,52 mg/L
	1c	0,47 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	14,4 mg/L
	2b	14,4 mg/L
	2c	13,90 mg/L
Sample 3: Process heating plant, bottom ash	3a	0,3 mg/L
	3b	0,22 mg/L
	3c	0,28 mg/L

Concentration of Copper Free L/S=8		
Sample1: Power plant 1, bottom ash	1a	0 mg/L
	1b	0 mg/L
	1c	0 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	9,2 mg/L
	2b	8,6 mg/L
	2c	9,3 mg/L
Sample 3: Process heating plant, bottom ash	3a	0 mg/L
	3b	0,02 mg/L
	3c	0 mg/L

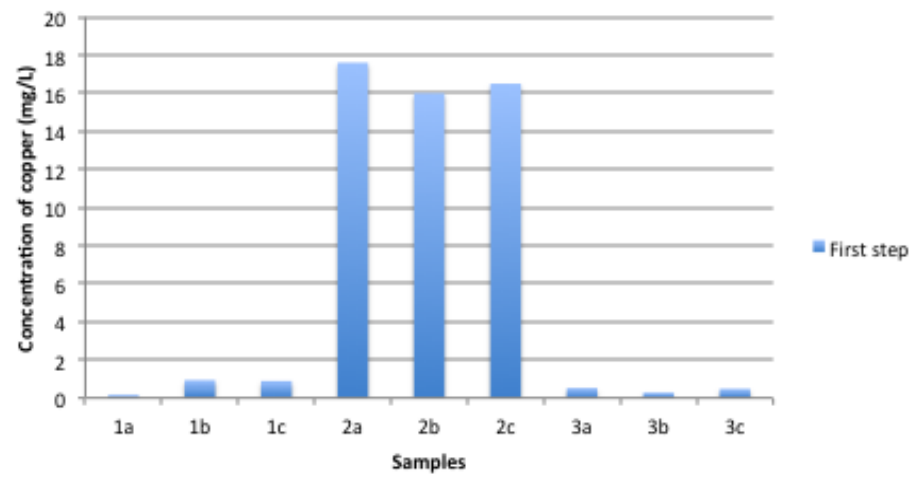
Concentration of Total Copper L/S=2		
Sample1: Power plant 1, bottom ash	1a	0,16 mg/L
	1b	0,94 mg/L
	1c	0,89 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	17,6 mg/L
	2b	16 mg/L
	2c	16,5 mg/L
Sample 3: Process heating plant , bottom ash	3a	0,52 mg/L
	3b	0,28 mg/L
	3c	0,48 mg/L

Concentration of Total Copper L/S=8		
Sample1: Power plant 1, bottom ash	1a	0 mg/L
	1b	0,02 mg/L
	1c	0,02 mg/L
Sample 2: Power plant 2, gasifier bottom ash	2a	10 mg/L
	2b	8,8 mg/L
	2c	9,4 mg/L
Sample 3: Process heating plant, bottom ash	3a	0,02 mg/L
	3b	0,03 mg/L
	3c	0 mg/L

APPENDIX 4



First leaching step



Second leaching step

