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# Behavior of Heavy Metals during the Agro-Industrial Wastes Gasification

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## ABSTRACT

The characterization analysis of three agro-industrial wastes was performed in order to study its thermal gasification. Some analyses such as determination of Ca, K and Mg concentration and determination of three representative toxic metals concentration Cd, Cr and Pb in all its oxidation states and the fundamental state, were carried out. The heavy metals concentration was also determined in the ashes obtained during the gasification process. The mobility of these elements was studied through three leaching tests. The behavior of heavy metals, sulfur and chlorine compounds, was predicted considering the presence of water vapor, syngas, Ca, Mg, K, Si, Al and other ash components. The heavy metals are not more concentrated in the gasification ash; these pollutants are released during this process. Ca, Mg and K presence in these residues would promote the pollutants retention. The ash of the studied waste can be disposed in controlled landfills or used in road construction, according to the obtained results during the leaching test DIN-DEV S4. The obtained results in the leaching test EPA 1311 TLCP classify these gasification ashes as no toxic waste.

**Keywords:** Gasification; Agro-Industrial Wastes; Ash

## 1. Introduction

The agro-industrial sector produces a significant environmental impact in specific geographical areas, due to generated waste, such as the Cuyo Region, Argentina. A strategy in this sense is to propose an appropriate agro-industrial waste management in order to minimize the emitted pollutants, transforming them into high value-added products or renewable energy source, tending to "Zero-waste".

During the 2011 harvest, 690,000 tons were used to produce wine, generating nearly 200,000 kg of stalks without considering other solid wastes, such as marcs and wine dregs. The latter are generally used for the by-products recovery.

However, reuse and/or disposal of exhausted marcs and wine dregs are a current problem in the region, because their disposal in landfills is not environmentally convenient due to they are not fully reused and large volumes are generated, requiring significant areas of land for their disposal. The waste from the fruits and vegetables canning industry have a high water content and, in many cases, significant amounts of lignocellulose materials. The final disposal in landfills is also performed in this region.

Moreover, there is a growing global interest in the technologies development for the exploitation of renewable energy sources because of environmental and economic reasons. In particular, due to the continuous increase in the cost of fossil energy resources, biomass is considered as one of the most promising and viable alternatives. Energy from waste is an important component of integrated waste management. One of the major limitations in the use of biomass wastes for energy production is its availability and moderate calorific value resulting in a low production and high costs compared to fossil fuels. The reduction of gases emissions, such as $SO_x$ and greenhouse gases; however, is agreed with the policies of current pollution control [1].

The energy conversion technologies and the biomass-based systems are the only electricity renewable source excluding hydro power, a crucial fact for future electricity production. A technology with a great future is the gasification. After more than 30 years of research, there is now worldwide interest in the use of $H_2$ as an alternative transportation fuel [2].

The steam gasification of waste is an attractive process for producing $H_2$-rich gas [3-5]. This process has been developed to reduce the amount of undesired products and the coke formation rate [6]. Furthermore, a vapor excess can easily be separated by condensation. Regarding existing gasification technologies, the fluidized bed is attractive because it provides a good contact between gas and solid, uniform temperatures and high reaction rates, compared to the fixed bed gasification [7]. Moreover, fluidized beds have a high flexibility in the feed in terms of shape, size and composition, as well as a wide range of operational and safety capabilities [8].

Considering the heavy metals, they are enriched in the solid waste of gasification (fly ash and bottom), and they are also released in the gas stream or tar. Their vaporization depends on the initial chemical speciation, gasification atmosphere, the fluid dynamics, the kinetics of heavy metals diffusion in the solid particles and reaction kinetics between the heavy metals and major components of ash [9,10].

The ash disposal conditions as well as their reuse are established by the trace elements concentration and their mobility [8].

In view of these aspects, the heavy metals behavior during agro-industrial waste gasification, their mobility out the ash matrix and the toxicity determination of generated solid waste during this process were studied.

## 2. Experimental

Agro-industrial residues from canning and wine sector were used: peach pits, stalks and marc, respectively. These industries are located in the province of San Juan, Argentina.

In order to obtain the ash, a differential reactor was used. It is constructed of AISI 316 stainless steel. It is constituted by a cylinder with 50 mm of diameter and 30 mm of length, heated by an electric resistance with electronic temperature control. **Figure 1** shows a used reactor scheme.

According to Kurkela *et al.* [11] (2006), for feeds with high alkali content, low gasification temperatures (T = 750˚C - 850˚C) and the steam addition are recommended to prevent the agglomeration in the fluidized reactor.

Skoulou *et al.* (2008) [12] studied the effect of temperature (T = 750˚C - 850˚C) and air equivalent ratio (ER = 0.2 - 0.4) in biomass gasification into a fluidized bed (ER is the ratio between the air sub-stoichiometric and the air required for complete combustion amounts). Experimental results showed that working at 750˚C and ER equal to 0.2, the $H_2$ optimal content in the syngas is obtained.

Taking into account these experimental results obtained by other researchers, the used gasifying agent was the steam and air mixture. The ER was equal to 0.2 and the temperature equal to 750˚C. The gasifying agent (air-steam mixture) entered from the reactor bottom. The syngas exited at the top.

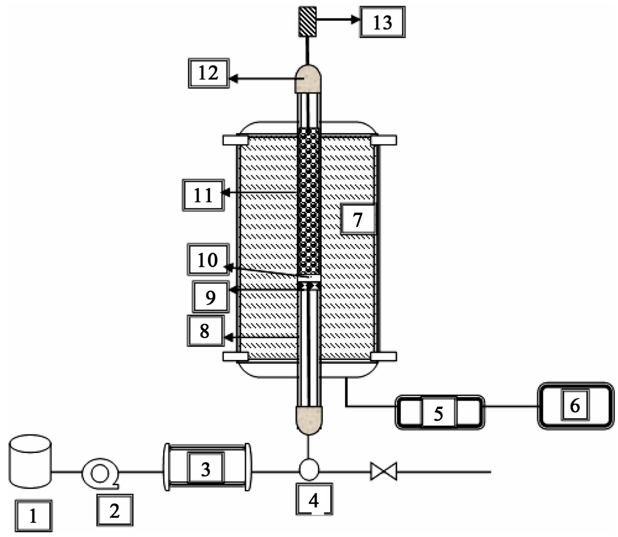


**Figure 1. Used reactor scheme. 1: water tank; 2: pump; 3: evaporator; 4: mixer; 5: power supply; 6: temperature controller; 7: electric resistance; 8: reactor; 9: support steel; 10: porous metal mesh; 11: agro-industrial wastes; 12: gas output; 13: thermocouple.**

For each test, the reactor is loaded with 50 to 60 g of agro-industrial wastes. The obtained ashes in this reactor are considered with similar characteristics as the ash bottom obtained in a fluidized bed reactor [13].

### 2.1. Agro-Industrial Wastes Characterization

The weight loss at 105˚C (ASTM D3173-87, Standard Test Method for Moisture in the Analysis Sample of Coal and Coke, 1996), the ash and the organic matter contents (ASTM D3172-89(2), Standard Practice for Proximate Analysis of Coal and Coke, 2002), the concentrations of Cd, Cr and Pb were determined for the studied agro-industrial wastes. In order to determine the heavy metal concentrations, first, the samples were digested according to EPA digestion (US Environmental Protection Agency, 1982). Then, the heavy metals concentrations were determined using a visible light spectrophotometer (HACH DR/2010 Spectrophotometer Datalogging portable).

Cd, Pb and Cr were considered due to their behaviors during heat treatments which are different. Cd vaporizes and it is not remained in the ash, Pb shows an intermediate behavior, and Cr is remained in solid residue of gasification [14].

In order to determine the influence of the presence of Ca, Mg and K in this biomass gasification, their concentration was determined in the studied agro-industrial wastes, using the atomic absorption method.

### 2.2. Heavy Metals Mobility of the Ash Mineral Matrix

With the purpose of study the heavy metals mobility of the ash mineral matrix, the Cd, Pb and Cr concentrations were determined in the solid wastes of gasification, using the analytical techniques described above. This study is very important because it determines the final disposal and/or further use.

Particularly, the use of the gasification ash in different applications contributes to the sustainability of biomass use in power generation. Several options are discussed: use as fertilizer, as a building material or as fuels [15]. The heavy metals mobility was studied by three different tests:

- The German test, DIN 38414 part 4 (DEV S4, German Standard Procedure for Water, Wastewater and Sediment Testing, 1984): It is used to classify the waste. The limits of heavy metals concentrations in the leaching solution are expressed in mg/l for disposal in landfill (Class 1) and for the ash use in road construction [16].
- The US EPA TCLP 1311 test (Toxicity Characteristic Leaching Procedure. Methods for Evaluating Solid Waste, 1992): It determines the potential leaching of organic and inorganic material in liquid, solid and multi-phase, of the residues in contact with groundwater. This test simulates landfill leaching conditions and the ash can be classified as toxic or not [17].
- The Dutch NEN 7341 test (Determination of the Leaching Behavior of Granular Materials: Availability Test, 1993): This test determines the maximum proportion of heavy metals leached from different wastes such as ash. This is achieved by leaching of finely milled solid (maximizing the contact surface) and using a high liquid/solid ratio. The metal fixation in the solid matrix is predicted with this test [18,19].

The DIN test uses the weaker leaching agent, distilled water, and the test NEN the stronger, nitric acid.

## 3. Results and Discussion

The results of the agro-industrial wastes characterization are shown in **Table 1**. The highest ash and water content were found in the stalk. A high water content increases the energy requirements to carry out the gasification, decreasing the efficiency of the plant, but on the other hand, it improves the synthesis gas quality by increasing the content of $CO_2$, $CH_4$ and $H_2$ [20] and decreasing hydrocarbons and tars levels. In order to optimize the gasifier operation, Pfeifer *et al.* [21] determined the optimum content equal 20% to 40% by weight at low temperatures heating.

Regarding the ash content, a low percentage of it will minimize the production of fly and the bottom ash. In general, these solids contain significant amounts of unreacted carbon and sulfur [22].

Cd, Cr and Pb are present in the composition of studied agro-industrial wastes. The stalks and the peach pits presented the highest Cd concentrations. With respect to Cr and Pb, the highest concentrations were found in winemaking waste.

**Table 1. Results of proximate analysis. Determination of heavy metals and Ca, K, and Mg concentrations in agro-industrial wastes.**

| | Stalk | Marc | Peach pits |
|---|---|---|---|
| **Weight loss at 105˚C (dry basis %)** | 73.23 | 55.06 | 35.57 |
| **Ash (dry basis %)** | 6.30 | 5.08 | 0.73 |
| **Organic matter (dry basis %)** | 93.7 | 94.92 | 99.27 |
| **Cd (mg/kg dry basis)** | 1.25 | 0.02 | 1.25 |
| **Cr (mg/kg dry basis)** | 25.00 | 37.50 | 3.125 |
| **Pb (mg/kg dry basis)** | 75.00 | 82.92 | 0.94 |
| **Ca (g/kg dry basis)** | 2.25 | 2.96 | 0.02 |
| **K (g/kg dry basis)** | 19.23 | 7.38 | 7.15 |
| **Mg (g/kg dry basis)** | 0.58 | 0.46 | 0.44 |

Considering the obtained results by analyzing the gasification ash (**Table 2**), the highest Cd and Pb concentrations were found in the marcs ash. For Cr, the highest concentrations were found in the stalks ash, in this case the metal concentrations is more variable (between 3.12 and 15.86 mg Cr/kg of dry weight waste).

Comparing the found heavy metals concentrations in the stalks and their ash, the Cd concentration variation is very small and the Cr and Pb concentrations of stalks are higher than these concentrations in their ash. In the case of the marcs and their ash, the Cd is more concentrated in the gasification solid waste, but the Cr and Pb concentrations are higher in the marcs. Comparing the concentrations of three heavy metals found in peach pits and their ash, a significant variation is not observed.

On this point, it is important to explain the heavy metals behavior during the biomass thermal treatment. When organic matter is consumed during any heat treatment, heavy metals are exposed to a hot and oxygen-depleted atmosphere, adjacent to the particle, presenting one of the following behaviors [9]:

1) They vaporize directly in the initial chemical species;

2) They react with a compound present in the atmosphere and then, they vaporize;

3) They remain unreacted in the mineral matrix.

The vaporized species enter in the gas flow where they react or condense. The condensed species form new particles (homogeneous nucleation) or they are deposited on the present particles surfaces (heterogeneous deposition). Homogeneous nucleation gas explains the substantial amount of very fine metal particles (diameter between 0.02 to 1 microns) found in the effluent gases. The heterogeneous deposition occurs in larger particles and they

 

**Table 2. Heavy metals concentrations in the ash.**

| Ash | Cd (mg/kg) | Cr (mg/kg) | Pb (mg/kg) |
|---|---|---|---|
| Stalk | 1.22 | 15.86 | 14.07 |
| Marc | 1.43 | 11.56 | 30.27 |
| Peach pits | 1.09 | 3.12 | 0.63 |

can be captured by the pollution control systems. To promote the heterogeneous deposition, it is necessary to limit the formation of fine metal particles.

The species formation with lower oxidation states than the initial states are favored by the reducing conditions. Furthermore, these metals may react with other released elements, as chlorine or sulfur. These new species are generally more volatile than the metal species present in the agro-industrial waste. The heavy metals volatilization during the gasification depends of their speciation and the gasification atmosphere.

Taking into account the heavy metals partition during gasification in fluidized bed reactor, the turbulence conditions during its operation cause a significant production of fly ash with high concentrations of these elements. The heavy metals partition during heat treatments in fluidized bed is governed by the fluid dynamics, the kinetics of heavy metals diffusion in the ash particles and reaction kinetics between the heavy metals and the ash components [10].

The chemical composition of the mineral matrix has a great influence on the kinetics of heavy metals vaporization; it determines the bonding strength between the mineral matrix and these elements, as well as the time required for diffusion out of the particle. Thus, basic species in the matrix ($SiO_2$, $Al_2O_3$, CaO) can react with these metals encapsulating them in the particle center [8]. The CdO (s), $Cr_2O_3$ (s) and PbO (s) may react with HCl, according to the following reaction:

$$\mathrm{MeO(s)+2HCl(g)\rightleftharpoons MeCl_2(g)+H_2O(g)} \qquad (1)$$

The used steam during the gasification phenomenon affects the reaction equilibrium and the heavy metals retention as oxides in produced solid waste [23]. A high water content of feed waste promotes this retention. Then, during the waste gasification using steam, the reaction is displaced to the left causing the formation of metal oxides. Notably, Mojtahedi and Salo [24] observed the presence of heavy metal chlorides in volatile phase when the gasification was carried out at high temperature.

According to Park *et al*. [25], MeO can react with the syngas according to the Reactions (2) and (3):

$$\mathrm{MeO(s)+H_2(g)\rightleftharpoons Me(s)+H_2O(g)} \qquad (2)$$

$$\mathrm{MeO(s)+CO(g)\rightleftharpoons MeC(s)+O_2(g)} \qquad (3)$$

when these reactions occur, the heavy metal gradually diffuses to the particle surface subsequently vaporized. These reactions can be inhibited by the addition of natural zeolite.

Vervaeke *et al.* [26] observed the augmentation of Cd and Pb concentrations in the fly ash comparing with bottom ash, during wood gasification in fixed bed; however, Cr remained in the bottom ash. Pinto *et al.* [22] detected higher Pb concentrations in the ash captured by cyclones comparing with bottom ash, confirming the above mentioned studies.

The amount of heavy metals which remains in the ash-decreases when the working temperature is high, close to 900˚C, and the synthesis gas quality increases.

According to Wei *et al.* [27], the sand, used as the bed material, adsorbs heavy metals, decreasing their concentrations in exit flow gas. The heavy metals release increases when the adsorption efficiency of this material decreases.

On the other hand, some metals such as Ca inhibit the bed material agglomeration, maintaining the fluidization quality and sand mixed with the biomass to be gasified. Then, Ca improves the fluidization delaying the heavy metals release [28,29].

Cui *et al.* [30] observed that most of the heavy metals are enriched in the exit gas flow. The experimental results are consistent with these observations. Approximately 70% of trace elements found in the synthesis gas, including three studied heavy metals, come from the gasified biomass and about 25% from gasification system.

Considering the alkali elements contents in the studied biomass, the Ca concentrations vary in a small range, except for the peach pits. For K, the found concentration in the stalk is very high compared to these concentrations in the marc and peach pits. The Mg concentrations in all analyzed residues vary in a small range.

If the gasification is carried out into a fluidized bed reactor, it is important to consider the biomass tendency to separate from the bed due to its low density, as well as the elutriation tendency of C small particle.

On the other hand, the gasification ash are inert and no involved in chemical equilibrium of the gasification reactions but, it may have a catalytic effect, accelerating the char gasification reaction with steam, especially when the ash contains metal oxides as $K_2O$, CaO, MgO, $P_2O_5$, etc. [31].

**Table 3** shows the principal conditions of lixiviation tests and relative ratio of the studied heavy metals found in the leaching solution.

Taking into account these results, the three metals were detected in leaching tests DIN-DEVS4, except to peach pits ash. Cd had higher mobility in leaching test DIN-DEVS4 for the marc ash. The Pb and Cr showed the highest mobility for the peach pits ash.

Considering the EPA-TLCP test results, all metals had the highest mobility in the case of the peach pits ash.

 

**Table 3. Main characteristics of the leaching tests. Relative ratio of the studied heavy metals found in the leaching solution.**

| Lixiviation test | Leaching solution | Time (h) | pH final value | Cd (%) | Cr (%) | Pb (%) |
|---|---|---|---|---|---|---|
| **DIN-DEV S4** | Distilled water | 24 | Final pH: 9 | | | |
| Stalk ash | | | | 66.64 | 1.50 | 2.75 |
| Marc ash | | | | 15.62 | 0.33 | 5.03 |
| Peach pits ash | | | | 0.00 | 85 | 100 |
| **EPA-TLCP 1311** | Acetic acid | 18 | Final pH: 4.5 | | | |
| Stalk ash | | | | 0.00 | 25,00 | 3,01 |
| Marc ash | | | | 15.62 | 6.67 | 4.04 |
| Peach pits ash | | | | 100 | 32.10 | 100 |
| **NEN 7341** | Nitric acid | 3 (step 1)<br>3 (step 2) | 7 (step 1)<br>4 (step 2) | | | |
| Stalk ash | | | | 33.36 | 24.00 | 3.86 |
| Marc ash | | | | 0.00 | 3.01 | 5.52 |
| Peach pits ash | | | | 0.00 | 100 | 100 |

Regarding NEN 7341 test results, Cd was not released from the peach pits and marcs ash, the Cr and Pb had highest mobility from the peach pits ash. Heavy metals mobility did not vary with the pH variation.

Established limit concentrations in the leachate solution for Cd by DIN 38 414 test are 0.05 and 0.005 mg/l to be placed in a landfill or used in the road construction, respectively. For Pb, these limit concentrations are 0.2 and 0.05 mg/l, respectively. The Cr is not regulated in this test. Taking into account the Cd and Pb concentrations in leachate solution from the studied wastes ash, it is concluded that these solid residues can be disposed in landfills or used in road construction.

The limit concentrations in the leachate solution, according to EPA test-TLCP 1311 are 1, 5 and 5 mg/l for Cd, Cr and Pb, respectively.

**Figure 2** show the relative ratio of the studied heavy metals found in the leaching solution for each tests.

In order to analyze the ash reuse as fertilizer, it is important to consider that the ash can be only K source, because they do not contain nitrogen and the phosphorus is content in forms with very poor solubility. The Mg and Ca content can improve quality especially in soil pH control. Considering this aspect and the retention of these elements in the solid matrix, it is concluded that the gasification ash from the stalks are most suitable for this use.

The ash from fluidized bed has constituent material of the bed (sand) and can be reused in road construction or concrete; however, its content of carbon, alkali and chlorine does not make it appropriate to be used as a construction material.

Considering the reuse as fuel, it is important to emphasize that the gasification ash, in general, have a significant amount of unburned carbon. This reuse is obviously the best choice because it has the same purpose as the original material: power generation, however, the heavy metals behavior must be considered [15].

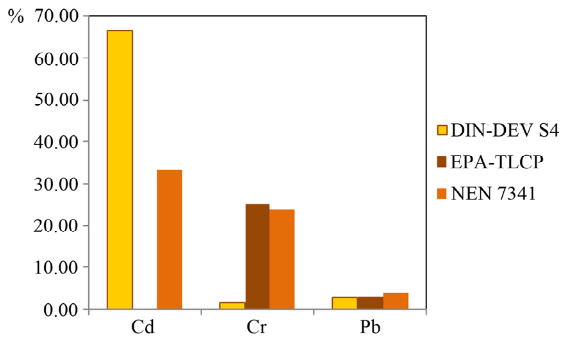


**Figure 2. Relative ratio of the studied heavy metals found in the leaching solution for stalk ash.**

## 4. Conclusions

The studied agro-industrial wastes have higher water content than 20%. Taking into account previous research, these wastes should be dried before the gasification in order to optimize the process yield. The residues analyzed have low ash contents. This aspect will have a significant impact on the obtained amount of fly and bottom ash from the gasification process.

The studied heavy metals are not more concentrated in the ash; therefore, they are released as gas, fly ash (homogeneous or heterogeneous nucleation) or tar.

The Ca content improves the retention of heavy metals. The improvement of the fluidization conditions produces their retention.

The results obtained in the leaching test DIN-DEV S4 suggest that the ash of this waste can be disposed in controlled landfills or used in road construction. It should be noted that the other parameters set by this test must be analyzed. The results obtained in the leaching test EPA 1311 TLCP are smaller than the limits set by the test.

The gasification ash can be reused for power generation or as fertilizers. In both cases, it will be necessary to perform an economic evaluation. The power generation reuse is most appropriate because it increases the gasification plant efficiency, but the heavy metals content in the ashes must be considered in order to minimize the environmental impact.

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