Analysis of the Pozzolanic Activity Potential of Hazelnut Husk Ash

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Abstract

Hazelnut husks are considered as a waste and therefore are discarded as fuel, compost or disposed into landfills. When used as fuel, ash is generated which can be used an additive for the construction industry in mortars or cement. Several studies have shown that different organic waste ash have pozzolanic properties and cand be used as additives. This study focuses on the determination of the pozzolanic potential of the ash from hazelnut husks, and the effect of the calcination temperature. For these purposes, a modification of the "Rapid determination of pozzolanic activity method" developed by Dalinaidu in 2007 was employed. The chemical principal of the methodology is based in the loss of the electro conductivity due to the reaction of the pozzolan with calcium hydroxide to form additional cementitious compounds. Because the particle size has an impact on the pozzolanic activity, initially the husk was shredded with a home-made blender and sieved though an ASTM Nº 40 sieved. Afterward the samples where calcinated at three different temperatures: 550º, 650º and 750ºC, crushed with a mortar and passed through the Nº 200 sieved. In this wat the particle size was ensured that was below 75 microns. Calcium hydroxide was employed for the reaction and the electroconductivity was measured though time until it stabilized. For each sample the electroconductivity was measured every 5 minutes until the measurement were stable. For the procedure it was determined that the total time that required was 50 minutes. The result showed that the electroconductivity between 550ºC and 750ºC had an increase from 0,24 mS/cm to 0,41 mS/cm. From the analysis all samples presented pozzolanic activity and the highest activity was observed with the sample that was calcinated at 750ºC. Therefore, hazelnut husk ash presents pozzolanic potential and temperature is a key factor in its potential as an additive in mortars or cement.

Keywords: *Chilean hazelnut, Pozzolanic activity, hazelnut shell, hazelnut husk ash, lime reactivity*

1. Introduction

Cement is a fundamental component in the construction and is a major manufactured materials and is the second most consumed material after water [1,2]. The production of cement is responsible for the 5-10% of the world CO2 emissions, contributing significantly to climate change [2,3,4]. As industry seeks innovative solutions to mitigate their environmental impact, several studies have explored the potential of residue biomass materials as a sustainable alternative in the production of cement or used as additives [4,5,6,7].

Chile is the fifth-largest producer of hazelnuts in the world and the largest producer in the Southern Hemisphere [8,9]. Hazelnut production is primarily concentrated in the Maule and Los Lagos regions. The area intended for cultivation of hazelnuts is approximately 36,395.61 hectares and a production of 65,646.73 tons is estimated [8,10]. The cultivation of hazelnuts generates significant quantities of waste, including leaves, skins, shells, husks and pruning materials [11]. Hazelnut husk is generated during the processing of the fruit and generates large quantities of biomass waste, that have no use and represent a disposal problem [11,12,13]. Therefore, using the husk as a renewable energy resource can be employed as an alternative disposal method [14]. As a subproduct of combustion, ash is generated, that can be used as an additive in cement, which can provide a sustainable approach.

Recent studies have shown that organic ash can exhibit favourable pozzolanic properties [15,16,17,18]. As a result, many studies have used organic waste ash as a partial replacement for cement [19]. For this purpose, hazelnut husk ash was calcinated at three different temperatures and then crushed and sifted to determine the pozzolanic potential and assess its performance based on its thermal activation temperature.

For this purpose, the determination of the pozzolanic activity is based on measuring electrical conductivity to assess pozzolanic activity in the aqueous suspension of pozzolan [20,21]. Pozzolans are siliceous and aluminous materials that react with calcium hydroxide when water is present. The pozzolanic reaction corresponds to a simple acid-base reaction between Ca(OH)2 and silicic acid H4SiO4 or SiO(OH4) [22]. Studies have shown that the pozzolanic activity can be measured through the variation of the electrical conductivity of suspensions obtained by mixing the ash and using a solution of calcium hydroxide [21].

2. Material and Methods

2.1 Materials and Sample preparation

The hazelnut husk corresponded to the European variety Corylus avellan, that were provided by local agricultural producer in Chile. Initially the husks underwent a size reduction process using a metallic household mill to ensure a small particle size. Afterwards the grinded husk was sieved though a 4-mesh ASTM sieve and all the material that did not pass was grinded again. The material that passed was sieve through a 200-mesh AST (particle size < 75 µm). This step was crucial to ensure uniform heat transfer during calcination and consistent reactivity during subsequent testing (See Fig 1).

The milled hazelnut husks were divided into three equal masses, and each sample was then subjected to calcination in a muffle furnace at specific temperature: 550ºC, 650ºC and 750ºC. The crucible employed in the calcination process had a ceramic led that avoided the generation of flames, that changed the temperature of the oven. Once the desire temperature was reached a minimum of

6 hours was maintained to ensure that all the mass of husk reached the set temperature. Afterwards samples where cooled in a desiccator, and the ash was re-sieved through the 200-mesh. All the samples were maintained in a desiccator, for not altering the weight through the absorption of moister.

Fig. 1. Particle before and after of grinding process

2.2 Thermogravimetric analysis of the Sample

A thermogravimetric analysis (TGA) was performed for the grinded hazelnut husk, utilizing a TA Instrument SDT Q600. This state-of-the-art equipment allowed for an in-depth exploration of the husk's thermal properties. Conducted under a controlled nitrogen environment at a steady flow rate of 50 ml/min, the analysis created optimal conditions for accurate measurements. The temperature range spanned from a cool 30ºC to a remarkable 900ºC, with a consistent ramp-up of 30ºC/min. This gradual heating process enabled a thorough investigation of the thermodynamic behavior of the hazelnut husk as it underwent transitions from moisture release to thermal decomposition.

The initial sample, weighing 23.5980 mg, was placed in a 90-microliter alumina crucible selected for its thermal stability and compatibility with thermogravimetric analysis (TGA). The resulting TGA curve is shown in Fig. 2. The curve reveals an initial weight loss due to intracellular water evaporation within the temperature range of 30°C to 150°C, corresponding to approximately 5.9% of the total sample mass. Subsequently, devolatilization commences, with the maximum rate of weight loss occurring between 260°C and 400°C. The weight loss associated with the devolatilization process beyond the moisture evaporation stage is approximately 51.7%.

Fig. 3 displays the results obtained differential thermogravimetric (DTG) curve. This analysis was used to determine the compositional characteristics of the lignocellulosic biomass. The decomposition temperature ranges for the main lignocellulose components are as follows: hemicellulose decomposes between 225°C and 325°C, cellulose between 250°C and 500°C, and lignin between 305°C and 375°C [23]. Additionally, it has been established that cellulose exhibits significantly higher reactivity compared to lignin, resulting in greater volatile production during thermal degradation [23]. Consequently, higher lignin content correlates with increased formation of carbonaceous residues, due to its lower thermal reactivity and degradation rate.

From the DTG curve (Fig. 4), three distinct peaks can be identified. The first peak corresponds to the loss of moisture and intracellular water. The second peak is associated with the degradation of hemicellulose, while the third peak reflects the decomposition of cellulose. Regarding lignin content, the analysis considered the temperature range from the end of the third peak up to 500°C. Based on this evaluation, the hemicellulose content was estimated to be approximately 27.7% by weight, cellulose at 25.9%, and lignin at 7.9%. The ash content considering 550ºC corresponded to 27,2%. Additionally, the DTG curve exhibits a smaller fourth peak between 715°C and 750°C, which can be attributed to the decomposition of calcium carbonate ($CaCO_3$) into calcium oxide (CaO).

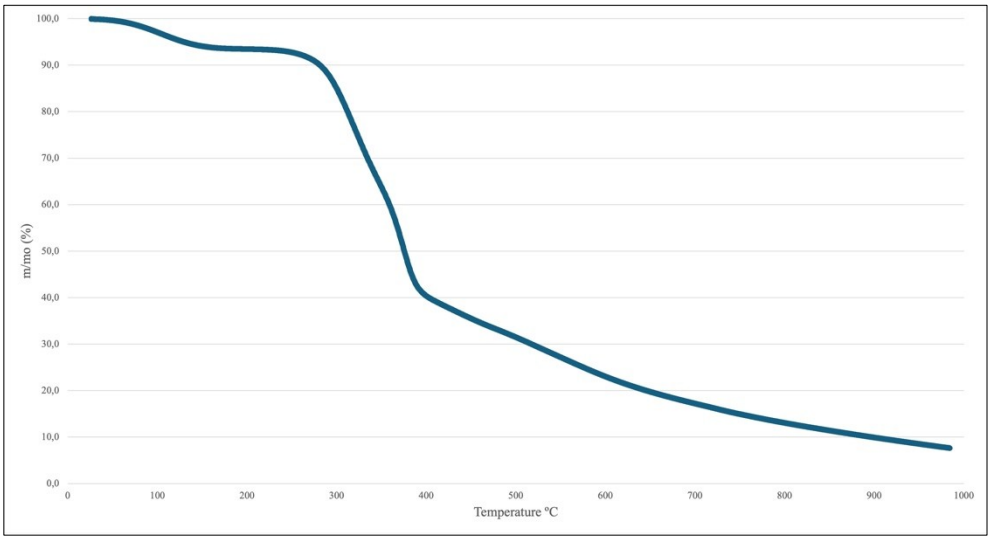


Fig. 2. Thermogravimetric analysis.

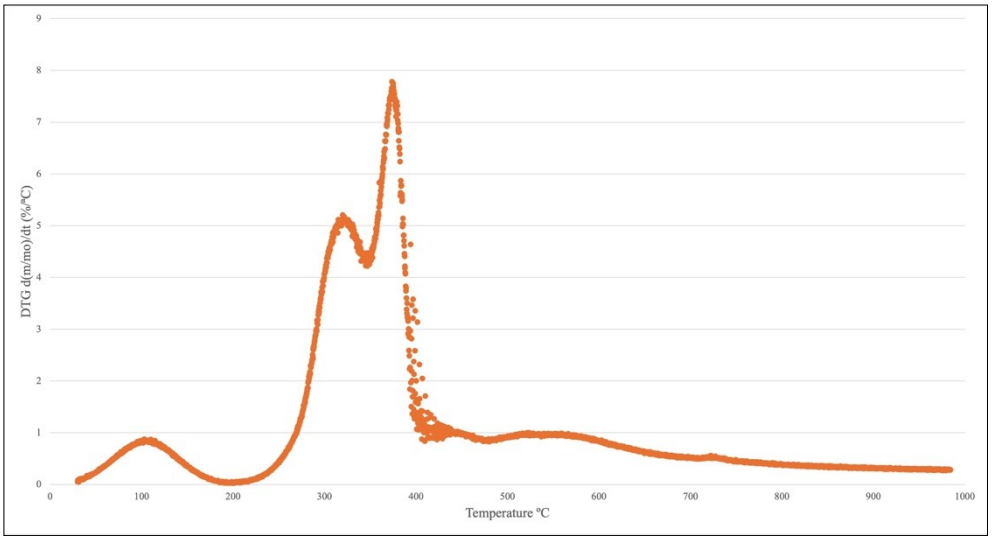


Fig. 3. DTG curve of Hazelnut.

2.3 Measuring of the pozzolanic activity

The evaluation of the pozzolanic activity was conducted through a systematic procedure to determine the saturation point of calcium hydroxide ($Ca(OH)_2$) solution and monitor its interaction with the natural pozzolanic material, specifically hazelnut husk ash. The initial step involved establishing a stable and precise measurement environment. To this end, all experiments were performed at a controlled room temperature of 25°C, ensuring consistent electroconductivity readings and minimizing temperature-induced variability.

Deionized water, with an initial electroconductivity of approximately 1.4 µS/cm which closely approximates the theoretical near zero value was used as the solvent medium. This initial electroconductivity was verified using a CDC401 Hach probe connected to an HQ440d multimeter, ensuring calibration accuracy. This setup aimed to accurately detect changes induced by the addition of calcium hydroxide and subsequent reactions with the hazelnut ash.

The experimental procedure commenced by filling a 250 mL glass beaker with 100 mL of deionized water. To facilitate uniform mixing and prevent localized concentrations, a magnetic stirrer bar was introduced, and the mixture was stirred continuously throughout the experiment. Precisely 0.025 grams of calcium hydroxide was carefully weighed using a high-precision analytic balance (KERN ABJ). This small quantity was added to the deionized water, and the mixture was allowed to sit undisturbed for approximately 90 seconds, or until the electroconductivity measurements stabilized, indicating equilibrium in calcium hydroxide dissolution.

Subsequently, incremental additions of calcium hydroxide powder were performed, with electroconductivity measurements recorded after each addition. These measurements continued until the electroconductivity value no longer increased with added calcium hydroxide, indicating saturation in the solution. The saturation point was identified at an addition of 0.3 grams of calcium hydroxide, which resulted in a stabilized electroconductivity of 9.31 µS/cm, representing the maximum ionic interaction capacity within the solution under the given conditions.

Following saturation determination, the pozzolanic activity assessment was carried out by preparing a series of suspensions with hazelnut husk ash. For each test, 2 grams of the ash were added to 100 mL of the saturated calcium hydroxide solution, maintaining a liquid-to-solid ratio of 50. Immediately after mixing, the initial electroconductivity of each suspension was recorded to establish a baseline. The suspensions were then subjected to electroconductivity measurements at 5-minute intervals over a 50-minute period, allowing for real-time monitoring of pozzolanic reactions, which are characterized by a gradual reduction in electroconductivity as calcium hydroxide reacts with the reactive constituents in the ash. The data obtained are summarized in Table 1, illustrating the kinetics and extent of the pozzolanic reactions under controlled laboratory conditions.

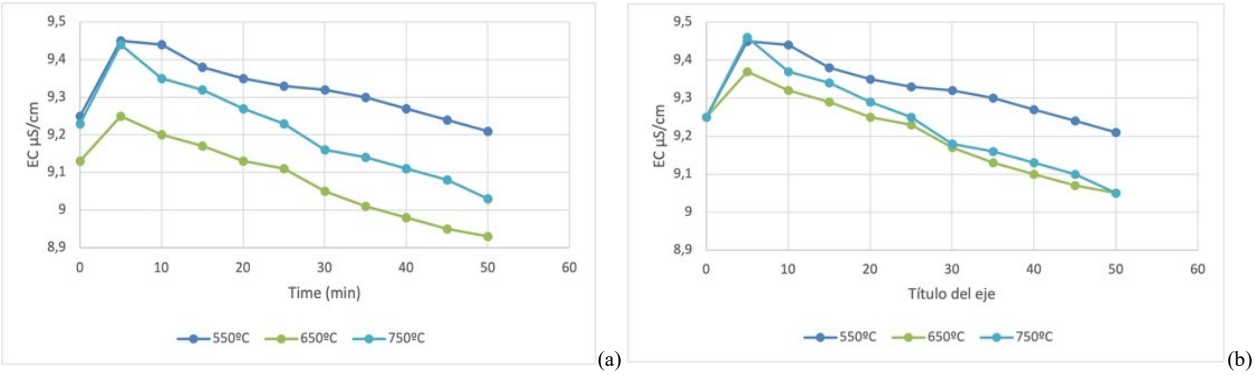
Tab. 1. Variation of the electroconductivity of the different hazelnut husk ash.

Time (min)	Sample 550ºC	Sample 650ºC	Sample 750ºC
0	9.25	9.13	9.23
5	9.45	9.25	9.44
10	9.44	9.2	9.35
15	9.38	9.17	9.32
20	9.35	9.13	9.27
25	9.33	9.11	9.23
30	9.32	9.05	9.16
35	9.3	9.01	9.14
40	9.27	8.98	9.11
45	9.24	8.95	9.08
50	9.21	8.93	9.03

At time 0 minutes, a small difference in electroconductivity was observed. To address this, the samples at 650ºC and 750ºC were adjusted to match the initial electroconductivity of the 550ºC sample (see Table 2 and Figure 3).

Tab. 2. Difference in electroconductivity between start and end in absolute.

Sample	Difference Between Start and End (µS/cm)
550ºC	0.24
650ºC	0.32
750ºC	0.41



(a) (b)

Fig. 4. Before and after of adjusting electroconductivity (a) original data, (b) centered data.

3. Conclusion

The thermogravimetric analysis of the lignocellulosic biomass revealed distinct thermal decomposition stages corresponding to moisture loss, and the degradation of hemicellulose, cellulose, and lignin. The sample's composition was quantified, indicating approximately 27.7% hemicellulose, 25.9% cellulose, and 7.9% lignin, with a significant ash content of 27.2%. Consequently, the percentage of ash that can be used as an additive should be no more that 27.7%.

The analysis shows a linear decrease in electroconductivity at all three temperatures, correlating with pozzolanic materials. At the 45-minute mark, the results indicate that 650°C exhibits higher pozzolanic activity than the other temperatures. However, at 50 minutes, both 650°C and 750°C yield similar results. Therefore, it is recommended to analyse this interval to determine the optimal temperature range.

The findings suggest that hazelnut husk has potential as an alternative additive or replacement in cement or mortar, and using hazelnut husk ash could help reduce carbon emissions in cement manufacturing.

The overall findings highlight the promising potential of hazelnut husk as an alternative additive or partial replacement for traditional materials in cement and mortar formulations. Utilizing hazelnut husk ash could contribute to reducing the carbon footprint of cement production, aligning with sustainable development goals and environmentally friendly construction practices.

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