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Title: FEASIBILITY OF USING OLIVE BIOMASS BOTTOM ASH IN THE SUB-BASES OF ROADS AND RURAL PATHS

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Corresponding Author: Professor Francisco Agrela, Ph.D.

Corresponding Author's Institution: Area of Construction Engineering, University of Cordoba, Cordoba, Spain

First Author: Manuel Cabrera, Ph. Dr.

Order of Authors: Manuel Cabrera, Ph. Dr. ; Julia Rosales, Architect and Civile Engineer; Jesús Ayuso, Ph. Dr.; José Estaire, Ph. Dr. ; Francisco Agrela, Ph.D.

Abstract: Clay soils are widely distributed throughout the world and are the source of multiple technical problems in their application for the construction of sub-grade and sub-road bases. These types of soils are found in areas where civilian infrastructure such as roads and rural roads must be built. Therefore, in many situations it is necessary to use stabilized expansive soils, in the formation of the foundation and structural layers of linear infrastructures. Soil stabilization is used to increase the load capacity of the soil, and mixtures of lime and cement are generally used as binders. In recent years, interest in the recycling of industrial products and byproducts has increased. One example of this is the use of biomass combustion in power plants. The management of significant amounts of waste (biomass bottom ash) from biomass power plants remains a problem. This paper presents the results of an experimental study for stabilizing expansive soil to determine its bearing capacity and mechanical properties via a triaxial test of the addition of biomass bottom ash. A double objective was targeted: reduction of the problems in using this type of soil and provision of a use for this type of waste. The results showed significant improvements in the mechanical. Therefore, herein is proposed the use of biomass bottom ash as a stabilizing agent for expansive soils, to improve the efficiency of the construction process by incorporating this product into a second life cycle as road bases.

1. Introduction

 In road and rural-path construction, it is essential to minimize the use of additional materials, and eliminate earth moving as much as possible, for environmental and technical considerations.

 The soil treatment techniques contribute to the competitiveness and sustainability of road engineering [1]. The engineering properties of construction materials determine their potential use and application in civil works. The material characteristics must satisfy the engineering functions that contribute to the durability and quality of the entire road structure [2]. Previous works have proved the feasibility of reusing industrial residues from different origin which have been applied in road construction [3-4].

 Soil stabilization is the process of alteration of geotechnical properties to satisfy engineering requirements [5]. Extensive studies have been carried out regarding the treatment of expansive soils using various additives, such as lime, cement, fly ash, industrial waste products, potassium nitrate, calcium chloride and phosphoric acid [6-13]. Traditional techniques of soil stabilization are often used to obtain geotechnical materials improved through the addition into soil of such cementing agents as Portland cement, lime, asphalt, etc. However, the traditional cementitious stabilizers like cement are under discussion, not only for their negative environmental effects during manufacture but also for their cost.

 Those types of additives have been used in soils to improve their engineering properties, and to modify physical and chemical reactions with soil elements in the presence of water [14-18]. For this reason, this work seeks the feasibility of using new by-products with pozzolanic characteristics for use as a soil stabilizer. Based on previous studies in which fly ash is used as a stabilizer due to its high CaO content, hydrates forming cementitious pozzolanic products similar to those formed during the hydration of Portland cement or lime. [19-21]. This research demonstrated the possibility of removing cement as a stabilizing material, replacing it with ash from the combustion of biomass for the generation of energy in combination with lime.

 In addition to cement, lime is one of the most used materials for soil treatment. Lime is a very caustic, pure white substance that results from the calcination of limestone. The common lime is calcium oxide CaO, also known as quicklime, which is widely used in construction.

 Lime can usually be obtained via thermal decomposition of materials such as limestone, which 68 contains calcium carbonate $(CaCO₃)$, extracted from sedimentary deposits called caliche. It is 69 subjected to very high temperatures (900–1200 \degree C), for a period of three days in a rotary kiln or in a special furnace called a lime kiln. However, if not managed, the process is reversible: while 71 cooling, the lime begins to absorb $CO₂$ from the air again; after a while, it once again becomes 72 CaCO_3 (calcium carbonate).

 The long-term operation of any construction project depends on the quality of the underlying soils. Unstable soils can create significant problems in built structures and pavements. With appropriate design and construction techniques, unstable soils can be chemically transformed into usable materials. In addition, the structural support provided by lime- or cement-stabilized soil can be exploited in pavement design.

 In general, the good results obtained from treatment with lime or cement, as applied in the construction of roads and esplanades, have extended this technique to any type of geotechnical problem and to esplanades with low bearing capacity [22]. The use of stabilized or treated soils, even with marginal and contaminated soils, avoids the reduction of natural resources by reducing the need for better quality soil. Moreover, clearance operations and transportation to a landfill are avoided, along with the extraction and transportation work conducted to replace the soil. It is a technique clearly focused on achieving greater sustainability.

 One of the biggest drawbacks of stabilization using lime or cement is their small particle size. Dust can be a problem, and its management is generally inadequate in populated areas. In addition to the high volumetric weight of such additives, which makes them more expensive to transfer, the dosage is altered in places where it is very windy. Moreover, the hydration process is more expensive when done in a plant rather than doing it at the site of application.

Biomass is a term with many definitions. For the purposes of this paper, biomass is considered as

any organic (non-fossil) material burned as fuel to generate electricity or produce heat.

 Biomass-based products produce solid residue (ash) a result of thermochemical degradation. Thermochemical processes include combustion, pyrolysis, and incineration of woody biomass.

 Currently, research is being conducted regarding the use of biomass ashes for civil works. In Spain, Andalusia leads in its scope of power generation from biomass, with 18 biomass combustion plants and a total installed capacity of 257.48 MW [23, 24]. The waste biomass in Andalusia from grapevines, olives, fruit trees, and poplar, is used as a source of renewable, sustainable energy to provide heat in homes. Biomass ashes are the solid by-products that remain after complete or incomplete combustion of organic matter. Industrial biomass ashes consist of biomass bottom ash (BBA), or slag, and biomass fly ash (BFA).

 BBA and BFA have been extensively studied, with focus on several applications. BFA has typically been used in agriculture due to its nutrient mineral content, including calcium, potassium and phosphorus [25]. Because of the increased production of this by-product, BFA has been investigated regarding its use in building materials. While fly ash utilization has been extensively studied, similar studies on the effective management and utilization of bottom ash have been scarce. BBA is traditionally disposed of in landfills.

 In recent studies, biomass bottom ash from wood combustion and agricultural olive residues was used as filler material in road embankments, as well as in the manufacture of cement-treated recycled materials and as additive in the manufacture of lightweight recycled concrete [26-28].

 Therefore, it would be interesting to study the possible application of bottom ash biomass for soil stabilization or treatment, and more specifically, for its use in the region of Andalusia in southern Spain. This region has problems related to expansive soils and has an abundance of European combustion power plants as well as higher concentrations of available biomass.

 The goal of the present work was to evaluate the possibility of using BBA as a soil treatment to stabilize the sub-bases of roads and rural paths according to the technical specifications for road works imposed by Spanish regulation [29].

 This article discusses the experimental results of improvement of the properties of an expansive soil when it is treated with biomass bottom ash. Thus, the treatment or stabilization of expansive

119 soils has been considered from the standpoint of civil engineering. These experiments have been 120 based on tests to evaluate the use of these types of soil as building materials.

 To these ends, the following parameters were measured to physically and mechanically characterise the samples: granulometric composition, absorption, density, compactability 123 according to the modified Proctor test, bearing capacity based on the CBR index, plasticity and the triaxial compression test, x-ray fluorescence spectrometry and scanning electron microscopy analysis with x-ray spectroscopy.

126 The potential for using BBA mixed with clays at certain percentages of dosage. This BBA 127 valorisation could avoid a large amount of the waste currently being sent to landfills, providing 128 economic and environmental incentives.

129

130 **2. Materials and methods**

131 *2.1 Biomass bottom ashes (BBA).*

132 In this work Olive Biomass Bottom Ash (BBA) was studied and applied in the formation of 133 granular materials to be applied in road structural layers.

134 Based on the data, a power plant burned approximately 40% olive cake and 60% wood biomass 135 (poplar, olive and pine).

- 136 The biomass sample analysed in this study was collected after combustion at the plant
- 137 BioLinares, as characterized in specific studies performed previously [26, 30]. A summary of the

138 physical and chemical characterization of this sample material is shown in Table 1.

139 **Table 1**: Summary of the main physical and chemical properties

 According to the results, BBA is composed of extremely porous particles with rough surface textures. The size of these particles varies from sand to fine gravel. The water absorption and saturated surface-particle density were measured. Absorption is an important factor to consider because many physical parameters of bottom ash are altered in the presence of excess water [31, 32]. Low dry-surface particle densities (SSD) were calculated for the BBA sample. Compared to traditional natural aggregates, low densities were obtained for BBA because it is composed of particles with low specific weight [33, 34]. The chemical composition of BBA indicated that 148 BBA primarily consists of Si, Ca and K, while the measured amounts of Mg, Fe, Al, Na and Ti (minor elements) were < 5%. Thus, Si is the most abundant element, followed by Ca and K (in similar amounts). Due to the nature of the material, the BBA sample tested contained 4.89% organic matter.

The X-Ray Diffraction Analysis (XRD) of the BBA (Figure 1) shows that the most representative

phase of the bottom ash is quartz, which is typically produced at high temperatures during the

combustion process. There is also significant presence of the crystalline phases of calcite, because

biomass fuel contains a naturally high content of wood waste.

165 *2.3 Expansive clay soil (ECS)*

 Expansive soils are those which show volumetric changes in response to changes in their moisture content. Such soils swell when the moisture content is increased and shrink when the moisture content is decreased. Consequently, expansive soils cause distress and damage to structures founded on them.

170 The expansive clay soil analysed was stockpiled on the premises of the University of Córdoba.

171 The maximum aggregate size was 2 mm. Chemical analysis highlighting the elemental content of

172 elements (Table 2) and the organic matter content was 1.20 wt%, as determined according to

- 173 standard UNE103 204.1993.
- 174
- 175 **Table 2:** Physical and chemical properties

| | | Quicklime | ECS |
|--|----|-----------|------------|
| Liquid limit (UNE 103-103-94) | | | 59.2 |
| Plastic limit (UNE 103-104-93) | | | 39.9 |
| Plasticity (LL-PL) | | | 26.3 |
| Organic matter $(\%)$ (UNE 103-204) | | | 1.2 |
| Elemental content $(\%)$ (UNE 80-215) | Si | < 0.01 | 23.48 |
| | Ca | 52.9 | 16.8 |
| | K | < 0.01 | 0.74 |
| | Mg | 0.15 | 1.17 |
| | Fe | 0.03 | 2.93 |
| | Al | 0.03 | 3.44 |
| | Na | 0.02 | 0.23 |
| | Ti | < 0.01 | 0.45 |

¹⁷⁶

178 Figure 2 shows the size distribution of the swelling clays. As a clay material, with much of the

179 sample tested, 95% were among the fractions below 0.063 mm.

180

¹⁷⁷

2.4 Mixtures

 In this study, four different percentages of BBA were added to the expansive material, 0, 15, 50 and 100%; furthermore, the expansive material was mixed with 5 wt% quicklime. Table 3 shows the mixes obtained in the laboratory as well as the name used for the different mixes. .

2.5 Methods

a) *Modified Proctor*

 The Modified Proctor compaction test is a laboratory geotechnical testing method used to determine soil compaction properties, specifically, to determine the optimal water content at which soil can reach its maximum dry density.

 The Modified Proctor compaction test, in accordance with UNE 103-501-94, consists of compacting soil samples with given water content in a standard mould with standard compaction energy. The procedure specifies a hammer weighing 4.5 kg and a freefall distance of 457 mm. All the materials were compacted in five layers, applying 60 blows to every layer.

b) California bearing ratio (CBR)

 This test method is used to evaluate the potential strength of sub-grade, sub-base and base course material, including recycled materials for use in road and airfield pavements. The CBR value

obtained in this test forms an integral part of several flexible pavement design methods.

This test is performed according to UNE 103 502-95, which describes the process for determining

the resistance index of soils called CBR. This index is not an intrinsic material value but depends

 on the conditions of density and soil moisture, as well as the overload to be applied while performing the test.

 This study was conducted with 25% Modified Proctor (MP), 50% MP and 100% MP value tests and an overload of 4.5 kg. The evolution of the tested specimens was examined under different external conditions and over time. The four test conditions were un-soaked CBR, 4-day soaked CBR, 90-day soaked CBR and 90-day in dry chamber CBR. The dry chamber had a temperature 213 of 20 \degree C and 72% humidity.

c) Triaxial compression test

 The Triaxial Compression Test is a laboratory test method used to assess the mechanical properties of rocks and fine-grained soils. It provides a measure of the confined compressive strength and the stress-strain characteristics of rock, soil or other material specimens. It is most often applied to soil and rock samples to simulate in situ confining pressures and to measure the corresponding strength and deformation characteristics. Triaxial compression tests performed over a range of confining pressures are used to define a material's strength envelope.

 The specimen (either rock or reconstituted soil) is encased by a thin rubber membrane and placed inside a pressure vessel. The pressure vessel allows the specimen to be loaded hydrostatically to the desired confining pressure, while the rubber membrane prevents confining fluid from contaminating the sample. In a conventional triaxial compression test, the specimen is first loaded hydrostatically to the desired confining pressure and then the axial load is increased to specimen failure while holding the confining pressure constant. The applied load and resulting deformation was measured using our data acquisition system to generate load-deformation curves. The sample was loaded until it:

- Exceeded its confined compressive strength (brittle failure), or

- Reached 15% axial strain.

 At sufficiently high confining pressures, the soils reach a brittle-ductile transition. Above this stress state, the material may continue to increase its load carrying capability without apparent failure as additional axial strain is imposed. In this case, the state of stress at an axial strain of 15% is conventionally used to define the strength envelope. For porous samples, a pore pressure can also be applied through a small hole beneath the specimen to simulate in situ conditions.

 The triaxial test is performed according to UNE 103 402-98, which determines the strength parameters of a material sample in CU test mode: consolidated and undrained, with a pore pressure measurement assay. The specimen is saturated, consolidated under isotropic conditions and the test proceeds until compressive failure.

d) Free-swelling

 A series of free-swell tests were conducted on specimens compacted with optimum water content and to a density equivalent to 100% of standard Proctor compaction. The apparatus used for free- swell testing was an odometer, according to UNE EN 103 601-96. The free swell is the increase in height (expressed in percent) when a specimen is laterally confined, subjected to a vertical pressure of 10 kPa and then flooded.

 The soil specimen was placed in a fixed-ring consolidation cell, and the specimen was subjected to a confining pressure. During testing, vertical movements of the specimen were monitored using a dial gauge and a linear variable differential transducer.

 After the specimen was placed in the apparatus and the seating load was applied, the height of the specimen was monitored. Once the height of the specimen came to equilibrium, data were logged from the linear variable differential transducer and water was added to the reservoir in which the soil specimen was sitting to begin swell testing the specimen.

 A total of five tests were made in parallel in the laboratory to evaluate the free-swell characteristics of all mixtures.

e) X-ray fluorescence spectrometry (EDXRF)

 The elemental concentrations were determined using energy dispersive an X-ray Fluorescence (EDXRF) Spectrometer according to UNE EN 196-2. EDXRF provides a rapid and non- destructive method for the analysis of trace and major elements in soil samples. Quantitative x- ray fluorescence data were collected using Panalytical NHM-X226 and quantified using Super Q software. The samples were fused and analysed by the Rietveld method.

265 *f) Scanning electron microscopy analysis with x-ray spectroscopy*

266 Analysis by Scanning Electron Microscopy (SEM analysis or SEM microscopy) was used for 267 solid material characterisation. SEM facilitates the study of particles and surfaces with the added 268 benefit of acquiring elemental composition for the sample being studied.

269

270 **3. Results and discussion**

271 *3.1 Modified Proctor*

 Figure 3 shows the graphical representation of the results of the Modified Proctor test, it is possible to observe that all the materials presented curves very insensitive to changes of moisture content, making it necessary to ensure that the moisture content was close to the optimum value during compaction.

 As expected, with the ECS+BBA mixture, the moisture and density values obtained were intermediate compared to those of the pure unmixed materials, highlighting the high humidity of these materials to achieve relatively low densities. In previous research [26], these results were confirmed for BBA. The plane curves shown indicate that these materials do not exhibit high sensitivity to changes in moisture for compaction.

Figure 3: Moisture-density relationships

283 *3.2 California bearing ratio (CBR)*

 Table 4 shows the results obtained from the CBR tests for the four modalities selected for these tests and for the materials studied, and Figure 4 shows graphs of the CBR values obtained for 100% MP in the four modes in which the tests were performed. For the 90-day soaked and 90-day dry chamber tests, the value represented is the average of three samples.

288 **Table 4**: CBR values

289

290 Comparing the values in the four test modalities it is observed that all $ECS + BBA$ mixtures 291 improved soil bearing capacity. The value compared to ECS was increased by 98% for ECS + 292 BBA (50/50) and by 64% for ECS + BBA (85/15) when un-soaked. The measured values in BBA 293 are consistent with the data obtained by previous authors [35], who characterized bottom ashes to 294 be applied in civil infrastructures.

 Previous research used ash from different byproducts such as Rice Husk Ash [36], Palm Oil Fuel Ash [37], 'Bagasse Ash' from sugar industry [38]. In all the researches the use of these ashes decreased CBR values (fundamental property of stabilized soil) or it has been necessary the use of cement to obtain acceptable conditions of stabilized soil. However, this new research demonstrates the high pozzolanic capacity of BBA due to its chemical composition and the viability of use as a stabilizing material.

309 The curves of axial deformation-deviatoric stress obtained in the tests can be observed in Figures

310 5, 6 and 7.

Figure 5: Stress-strain curves: Tests performed with 650 (Kpa) confining pressures

Figure 6: Stress-strain curves: Tests performed with 750 (Kpa) confining pressures

Figure 7: Stress-strain curves: Tests performed with 850 (Kpa) confining pressures

 A uniform mass of expansive soil which becomes saturated with moisture will exert pressure in all directions as each individual expanding clay mineral seeks to occupy more space. The direction and magnitude of soil movement will depend upon the magnitude of the confining pressure at any particular point of resistance. Soil movement will be minimized where confining pressures are largest, while movement will be greatest where the magnitude of the confining pressure is smallest.

- The analysis of the stress-strain curves obtained in the triaxial tests makes it possible to highlight the following aspects.
- In the tests conducted with BBA, the deviatoric stress obtained was much greater than that obtained with ECS.
- Once BBA was mixed with ECS in the proportion 50:50 wt%, the deviatoric stress was reduced

by approximately 55% with respect to the values obtained with BBA.

 The mixing of ECS with BBA produced a deviatoric stress approximately 35% of the value obtained with BBA.

- The interpretation of those curves allowed us to calculate Mohr-Coulomb parameters with the aid
- of the s´-t diagrams, as seen in Figure 8.

 Figure 8: Tests performed with ECS and BBA (Left) and tests performed with ECS + BBA (50/50) and ECS + BBA (85/15) (Right).

 It can be observed that the relevant points fit very well on lines that can be interpreted as representative of the Mohr-Coulomb failure criteria. The values of friction angle obtained with these tests are summarized in Table 6.

| 341 | | | | Table 6: Triaxial strength parameter values | |
|-----|--|--|--|---|--|
|-----|--|--|--|---|--|

| | Strength Parameters (Effective) | | |
|------------------|--|--|--|
| | Φ' | | |
| ECS | 34.6 | | |
| BBA | 43.7 | | |
| $ECS+5%$ QL | 27.5 | | |
| $ECS+BBA(50/50)$ | 40.7 | | |
| $ECS+BBA(85/15)$ | 31.9 | | |
| | | | |

Table 6 shows the values of the Mohr-Coulomb failure criteria obtained in the tests.

All the materials seem to exhibit non-cohesive behaviour. ECS has a friction angle of 35° (a bit

large for its clayish nature) that increases to 41° once mixed with 50% BBA. The mix of 85%

ECS with 15% BBA seems to have strength similar to that of the original ECS.

From this point of view, the strength of all of these materials, and their combinations, can be

considered high and sufficient to build any type of embankment.

3.4 Free-swelling

Furthermore, upon completion of the CBR tests (Section 3.2), the expansivity swelling values of

each material (expressed in percent) were obtained. Table 7 shows the results obtained for the

various swelling tests conducted.

Table 7: Swelling test values at four days

 As shown in Table 7, the ECS sample showed a high percentage of free swelling. When the ECS was mixed with 50% bottom ash from the biomass (i.e. ECS + BBA at 50/50), the free swelling was reduced by 99.5%. This reduction was similar to that achieved with the mixture 359 manufactured with 5% lime (ECS $OL + 5%$).

Regarding CBR swelling, the behaviour patterns were similar to those shown for free swelling. It

can be concluded that the use of BBA reduces the expansion of expansive soils to the same extent

(percentage) as lime. This demonstrates economic and environmental benefits from using this

industrial by-product in this way.

3.5 X-ray fluorescence spectrometry (EDXRF)

Using Quantitative X-ray Fluorescence Analysis, the different types of experimental materials

and processes were analysed to compare their predominant elements, shown in Table 8.

370 According to the results, this BBA contains a mixture typical of olive waste ash, but with higher 371 values of potassium (8.97%). Also, the increase of lime in BBA, as shown in Table 8, can lead to 372 increased pozzolanic activity [39].

373

374 *3.6 Scanning electron microscopy analysis with x-ray spectroscopy*

 By this procedure the signals generated during the analysis produce two-dimensional images which reveal information about the tested samples (see charts in Figure 9 including external morphology: texture). The present study also includes the composition of samples as estimated by x-ray spectroscopy.

 Figure 9: Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). The microstructure of samples was characterized using SEM. In Figure 9, it was observed that the BBA particles were irregular in shape. The ECS particle size was typically < 63 μm and in agreement with the particle size analysis results.

 The expansion potential of any particular expansive soil is determined by the percentage of clay and the type of clay in the soil. The clay particles which cause a soil to be expansive are extremely small. Their shape is determined by the arrangement of their constituent atoms, which form thin clay crystals.

- According to EDX analysis with the SEM, the clays belong to a family of minerals called silicates. The principal elements in the clay were silicone, aluminium and oxygen; whereas the major elements in the BBA were Ca, K, Si and Al (Table 9).
- **Table 9:** Percentage weight of elements

4. Conclusions

Based on the results obtained, we present the following conclusions:

 - The addition of BBA in all the mixtures improved the bearing capacity, mainly due to the high calcium content that increased the pozzolanic activity.

401 - When the ECS was mixed with 50% bottom ash from biomass, the free swelling was reduced by 99.5%. This result is similar to those reported for the mix with 5% lime.

403 - The flat curves obtained by the modified Proctor test confirm that BBA do not exhibit high sensitivity to changes in moisture for compaction.

- Regarding the values of Mohr-Coulomb failure criteria obtained in the tests, the strength of all of these materials (and their combinations) can be considered high and sufficient to build any type of embankment.

 - It can be concluded that the use of bottom ash from biomass combustion reduces the expansion of expansive soils to the same extent as from treatment with lime.

 The present work has proved, at least for certain dose percentages, the benefits of BBA for improved mechanical ability and stabilization when used for material construction in civil infrastructure. The valorisation of this product, instead of exploiting natural resources or non- renewable natural resources, can eliminate the negative impact associated with the indiscriminate disposal of this by-product in landfills.

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