Sustainable management of oil refining sludge by means of geopolymers production


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1. Introduction
2. Experimental
3. Results
4. Conclusions
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Portland Cement Production

Environmental problems

- Clinker production requires high temperatures (1450°C)
- 5-7% of global CO₂ emissions
- Extraction of raw materials
Alternative to Portland cement?
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Industrial waste

Environmental problems

- Huge amount of waste accumulates in landfills
- No chance of being reused

Oil refining sludge
Chamotte
Rice husk ash
Other use or application for industrial waste?
Sustainable management of oil refining sludge by means of geopolymers production

Alternative to Portland cement?

Another use or application for industrial waste?

Geopolymers

Resulting from
- Are Inorganic aluminosilicates
- 3D Amorphous Structure

Advantages
- Cementitious properties comparable to PC.
- Low emissions of CO$_2$
- Excellent properties:
  - Durability
  - Stability
  - Mechanical resistance

Reaction of Geopolymerization
It is necessary

1. PRECURSOR
Material composed of Al and Si

2. ACTIVATOR
Alkaline solution

Chemical reaction with low T$_a$

Form tetrahedral units (Td)

Setting and hardening

Structure formed by polymer chains

$M_n((SiO_2)z(AlO_2))_n * wH_2O$
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The main objective of this study is the development of new geopolymeric materials using water treatment sludge from the oil refining industry as a raw material, in order to valorise a new type of raw material that has not been used in the production of these materials.
1. Raw Material

Andalucía

There are 1.65 million hectares of olive groves

Season

4 million tonnes of olives are produced in an average olive season

Olive oil

Of this, around 3.7 million t/year are used for the production of olive oil
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1. Raw Material

From olive oil production

Waste

Oil sludge

Raw material for the production of geopolymers
Sustainable management of oil refining sludge by means of geopolymers production

2. XRF of Raw Material

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>Na₂O</th>
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<tbody>
<tr>
<td>Oil Sludge (OS)</td>
<td>2.11</td>
<td>53.55</td>
<td>0.710</td>
<td>0.759</td>
<td>0.311</td>
<td>6.29</td>
<td>0.358</td>
<td>3.70</td>
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<tr>
<td>Chamotte (CH)</td>
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<td>12.11</td>
<td>4.67</td>
<td>8.67</td>
<td>1.88</td>
<td>0.471</td>
<td>3.25</td>
<td>3.60</td>
</tr>
<tr>
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<td>0.144</td>
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Table 1. Chemical composition (XRF) of raw materials

Oil sludge should be combined with silica-rich wastes.
2. XRF of Raw Material

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<th>Raw Material</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
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Table 1. Chemical composition (XRF) of raw materials

Oil sludge should be combined with silica-rich wastes. Alumina-rich waste should be combined with silica-rich wastes.
3. Preparation of Raw Materials

1. Dry
2. Calcine (if necessary)
3. Crush
4. Grind
5. Sieve (0.100 mm)

The process takes weeks

Wastes used as raw materials

- Oil sludge
- Chamotte
- Rice husk ash
4. Preliminary studies for the production of geopolymers

**Precursors**
- 100% Oil Sludge (OS)
- 80% Oil Sludge (OS) + 20% Rice husk ash (RHA)
- 80% Oil Sludge (OS) + 20% Chamotte (CH)

**Activator: commercial solution**
- Solution of 100% NaOH
- Solution of NaOH and Na$_2$SiO$_3$ at 50%.

**Compressive Strength**

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<th>28 days (MPa)</th>
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Problems with the commercial activator

- Low mechanical strength
- Specimens break in contact with water
5. Manufacture of geopolymers with alternative activator

Preparation of the alternative activator

1. A 10M NaOH solution is mixed with diatomites
2. 6 hours in the reactor with stirring at 80°C.
3. Vacuum filtration
4. Alternative activator
The first mixture was 100% of OS, (which could not be produced because the mixture was cracked) then the OS was substituted at different percentages by the precursor RHA or CH: 5%, 10%, 15%, and 20% (by weight).

### Table 2. Mix proportions for assessed simples.

<table>
<thead>
<tr>
<th>Mix</th>
<th>OS (g)</th>
<th>RHA (g)</th>
<th>Relation L/S</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% OS</td>
<td>150</td>
<td>0</td>
<td>X</td>
</tr>
<tr>
<td>5% OS</td>
<td>7.5</td>
<td>142.5</td>
<td>1.55</td>
</tr>
<tr>
<td>10% OS</td>
<td>15</td>
<td>135</td>
<td>1.55</td>
</tr>
<tr>
<td>15% OS</td>
<td>22.5</td>
<td>127.5</td>
<td>1.55</td>
</tr>
<tr>
<td>20% OS</td>
<td>30</td>
<td>120</td>
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7. Manufacture of geopolymers with alternative activator

Precursors + Activating solution → Mixer → Striking table → Moulds → Specimens

Activating solution:
- 5% OS
- 10% OS
- 15% OS
- 20% OS
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8. FTIR of Raw materials

- **Chamotte**
  - CO$_3^{2-}$ groups
  - Si-O-Si/Al
  - O-Si-O

- **RHA**
  - O-H
  - Si-O-Si
  - O-Si-O

- **Oil sludge**
  - O-H
  - Si-O-Si
  - O-Si-O
8. FTIR of Raw materials

FTIR of Oil sludge

- Stretching of vibrations from $O-H$
- $O-H$ bending ($H_2O$)
- Asymmetry $Si-O-Si$
- $O-Si-O$ bending vibration characteristic of silica
8. FTIR of Raw materials

FTIR of RHA

- **O–H bending (H₂O)**
- **Si–O–Si stretching quartz**
- **Asymmetrical stretching vibration of Si–O–Si**
- **O–Si–O bending (SiO₄)**

**Wavenumber (cm⁻¹)**

**Transmittance (%)**

<table>
<thead>
<tr>
<th>Wavenumber (cm⁻¹)</th>
<th>Transmittance (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3900</td>
<td></td>
</tr>
<tr>
<td>3400</td>
<td></td>
</tr>
<tr>
<td>2900</td>
<td></td>
</tr>
<tr>
<td>2400</td>
<td></td>
</tr>
<tr>
<td>1900</td>
<td></td>
</tr>
<tr>
<td>1400</td>
<td></td>
</tr>
<tr>
<td>900</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td></td>
</tr>
</tbody>
</table>

- Transmittance is shown as a percentage on the y-axis.
- Wavenumber is shown on the x-axis.

**8. FTIR of Raw materials**

- The figure shows the FTIR spectrum of RHA (palm oil refinery ash) with key absorption peaks labeled.
- The spectrum includes wavenumbers and transmittance values for identifying chemical bonds and their vibrations.

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8. FTIR of Raw materials

FTIR of Chamotte

This band suggests the presence of $\text{O-C-O}$ bonds of $\text{CO}_3^{2-}$ groups associated with carbonate phases.

Asymmetrical stretching of $\text{Si-O-T}$, being T Si or Al tetrahedral.

Bending vibrations of $\text{O-Si-O}$.
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9. XDR Raw materials

XRD Chamotte Waste

XRD Oil sludge

XRD RHA

Very amorphous
10. Mechanical and physical tests

**Compressive Strength**

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<tbody>
<tr>
<td>RHA 7d</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RHA 28d</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CH 7d</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CH 28d</td>
<td></td>
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**Flexural Strength**

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- Higher strengths are achieved with RHA residue.
- Maximum strengths are achieved at 28 days of curing with 20% oil sludge (OS), reaching 36.6 MPa with the RHA residue, while 15.7 MPa is achieved with the CH residue.
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- Flexural strength increases with higher percentages of oil sludge.

- Higher strengths are achieved with the CH residue.

- The maximums are achieved at 28 days of curing with 20% oil sludge, reaching 12.6 MPa with the CH residue, while 8.87 MPa is reached with the RHA residue.
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**Bulk density (kg/m³)**

- 5% OS
- 10% OS
- 15% OS
- 20% OS

**Water absorption (%)**

- 5% OS
- 10% OS
- 15% OS
- 20% OS

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The test specimens with 5% and 10% oil sludge with the RHA residue were broken in water.

- Bulk density is similar, reaching maximums with 20% oil sludge.
- The lowest water absorption is reached at 28 days of curing with 20% oil sludge.
- The lowest absorption occurs with the RHA residue.
- Higher compressive strengths produce higher densities and lower water absorption.
• The test specimens with 5% and 10% oil sludge with the RHA residue were broken in water.

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Apparent porosity (%)

Total porosity (%)

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The apparent porosity decreases with increasing percentage of oil sludge.

This decrease is most noticeable after 28 days of curing.

The RHA residue test specimens with oil sludge (percentages higher than 10%) show a lower apparent porosity.

The test specimens with the lowest apparent porosity are those made with 20% oil sludge.
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- Porosities are lower with 20% oil sludge and similar with both wastes, RHA and CH.
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**Apparent porosity (%)**

- **Compressive strengths**
- **Water absorption**
- **Porosity**

**Total porosity (%)**

- **Water absorption**
- **Porosity**
Sustainable management of oil refining sludge by means of geopolymers production

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9th International Conference on Sustainable Solid Waste Management 15-18 June. Corfu 2022
Conclusions

These studies have shown that the recovery of oil sludge is possible through the manufacture of new geopolymeric materials, as their chemical characterisation indicates that they have a high alumina content, for this, it is necessary to mix them with other materials that have a high silica content. In this way, it is possible to recover these by-products, giving them a new use, bringing us closer to the circular economy.

Replacing oil sludge (OS) with RHA or CH improves the mechanical and physical properties of 100% OS. Promising physical and mechanical characteristics have been obtained.

The alkaline activator used does not contain silicate (the cause of most geopolymer contamination and its economic cost).

On the one hand, it has been proven that better mechanical and physical properties are obtained with the RHA residue, as long as the presence of OS is in a percentage higher than 10%, reaching compressive strengths of 36.6 MPa. On the other hand, although lower strengths are obtained with the CH residue, it can be combined with low percentages of oil sludge without the test specimens breaking in water.

This is a good environmental solution, as it is possible to develop an economical and sustainable material thanks to the use of industrial by-products.
Thank you!

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ACKNOWLEDGMENTS

This work has been funded by the project Activalo2: Valorisation of Urban and Industrial Wastewater Treatment Sludge in the Manufacture of New Sustainable Alkaline Activated Materials for a Circular Economy (UJA-1380933) Proyectos de I+D+i en el marco del Programa Operativo FEDER Andalucía 2014-2020.