Improvement of biofilm formation in trickle bed reactors by surface modification of different packing materials

AUTEX

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**Introduction**

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under Grant Agreement no. 761042 (BIOCON-CO₂). This output reflects the views only of the author(s), and the European Union cannot be held responsible for any use which may be made of the information contained therein.

**STAGE 1**
- **Gas out**
- **Liquid in**
- **Water in**
- **Liquid distributor**
- **Packing**
- **Cooling jacket**
- **Gas in**
- **Liquid out**

**Trickle bed reactor**

**STAGE 2**
- **Cupriavidus necator**
- **3-Hydroxypropionic acid**

**TARGET COMPOUND**

**STAGE 3**
- **Acrylic deriv. (Biopolymer)**

- **To avoid overexploitation of natural resources**
- **To reduce GHG (Greenhouse Gases) emissions**
- **To find alternatives to currently used petroleum-based materials**
Packing materials in TBR

Parameters influencing the attachment, growth and biofilm formation:

❖ Electrostatic interaction between support and bacteria
❖ Surface area and surface roughness of the support
❖ Size and shape of the bacteria
❖ Hydrophobic or hydrophilic nature of the support and bacteria
❖ Availability of nutrients
❖ Shear forces in the bioreactor

RASCHIG RINGS – CONVENTIONAL PACKING MATERIAL

❖ Provide a large surface area within the reactor
❖ Random packing
❖ High economic cost
The aim of this research is to study innovative, efficient, environmentally friendly and low-cost packing materials, by analyzing their characteristics, bio-adhesion properties and growth of bacteria.

**HARDWOOD CHIPS**
- Hardwood chips have vessels, higher density and higher concentration of lignin compared to softwood.
- Most types of bacteria are not able to degrade lignin.
- High surface energy, roughness and porosity.

**POLYURETHANE FOAMS**
- Reticular foam plastics, such as polyurethane foams, present a high porosity and large surface area.
Advanced packing materials

FIBROUS MATERIALS (PES TEXTILES)
- Three different configurations
- Large surface area and porosity

POLYISOCYANURATE (PIR) BASED FOAM
- Thermoset plastic
- Large surface area and porosity

POLYPROPYLENE PELLETS
- Have been found to provide good adhesion and biofilm growth properties
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## Wettability

### Tensiometer - Wilhelmy method
- WCA>90° - hydrophobic surface
- WCA<90° - hydrophilic surface

<table>
<thead>
<tr>
<th>Category</th>
<th>Contact angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raschig rings</td>
<td>58.2 ± 4.3°</td>
</tr>
<tr>
<td>Beech wood chips</td>
<td>64.1 ± 4.9°</td>
</tr>
<tr>
<td>Polyester 3D fabric</td>
<td>65.8 ± 8.6°</td>
</tr>
<tr>
<td>Polyester woven fabric</td>
<td>66.6 ± 0.5°</td>
</tr>
<tr>
<td>Polyester nonwoven</td>
<td>66.6 ± 0.5°</td>
</tr>
<tr>
<td>Eucalyptus wood chips</td>
<td>88.2 ± 3.7°</td>
</tr>
<tr>
<td>Soft polyether-polyurethane foam</td>
<td>88.8 ± 0.2°</td>
</tr>
<tr>
<td>Hard polyether-polyurethane foam</td>
<td>90.7 ± 1.7°</td>
</tr>
</tbody>
</table>

Good wetting

Bad wetting
Morphology (SEM)

Raschig rings

Beech wood

Eucalyptus wood
Morphology (SEM)

**Foams**

<table>
<thead>
<tr>
<th>Hard PU</th>
<th>Soft PU</th>
<th>Polyisocyanurate</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Hard PU" /></td>
<td><img src="image2" alt="Soft PU" /></td>
<td><img src="image3" alt="Polyisocyanurate" /></td>
</tr>
</tbody>
</table>

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Morphology (SEM)

Polyester fabrics

Woven

Non-woven

3D

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### Apparent density

**Apparent density** = \( \frac{\text{Material weight}}{\text{Rectort volume} - \text{Material volume}} \)

<table>
<thead>
<tr>
<th>Category</th>
<th>Apparent density (g/cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raschig rings</td>
<td>2.58</td>
</tr>
<tr>
<td>PES 3D fabric</td>
<td>2.09</td>
</tr>
<tr>
<td>PES woven fabric</td>
<td>1.16</td>
</tr>
<tr>
<td>PES nonwoven</td>
<td>1.00</td>
</tr>
<tr>
<td>Eucalyptus wood</td>
<td>0.99</td>
</tr>
<tr>
<td>PP pellets</td>
<td>0.92</td>
</tr>
<tr>
<td>Beech wood</td>
<td>0.77</td>
</tr>
<tr>
<td>Soft PU foam</td>
<td>0.098</td>
</tr>
<tr>
<td>Hard PU foam</td>
<td>0.072</td>
</tr>
<tr>
<td>PIR-based foam</td>
<td>0.032</td>
</tr>
</tbody>
</table>
Bacterial adhesion and growth

Bacterial colonisation essay with potential advanced packing materials

1. Incubation of bacteria at 30°C with rotational shaking during 24 hours.
2. Washing of the packing materials to remove non-adhered bacteria.
3. Recovery and quantification of the cells forming the biofilm.
Bacterial adhesion and growth

<table>
<thead>
<tr>
<th>Material</th>
<th>CFU·cm⁻³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raschig rings</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>Beech wood</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>Eucalyptus wood</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>Soft PU foam</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>Hard PU foam</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>PIR foam</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>PP pellets</td>
<td>1.0 x 10⁸</td>
</tr>
<tr>
<td>PES woven textile</td>
<td>1.0 x 10¹⁰</td>
</tr>
<tr>
<td>PES nonwoven</td>
<td>1.0 x 10¹⁰</td>
</tr>
<tr>
<td>PES 3D textile</td>
<td>1.0 x 10¹⁰</td>
</tr>
</tbody>
</table>
Bacterial adhesion and growth
Behaviour upon water submersion

PES nonwoven

Before

After

PES woven

Before

After

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## Costs

<table>
<thead>
<tr>
<th>Category</th>
<th>Price (€/kg)</th>
<th>Apparent price (€/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raschig rings</td>
<td>680</td>
<td>263</td>
</tr>
<tr>
<td>PIR-based foam</td>
<td>70</td>
<td>2333</td>
</tr>
<tr>
<td>Hard PU foam</td>
<td>53</td>
<td>757</td>
</tr>
<tr>
<td>Soft PU foam</td>
<td>53</td>
<td>530</td>
</tr>
<tr>
<td>PES 3D textile</td>
<td>16</td>
<td>7.7</td>
</tr>
<tr>
<td>PES woven textile</td>
<td>15</td>
<td>13</td>
</tr>
<tr>
<td>PES nonwoven</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Eucalyptus Wood</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>PP pellets</td>
<td>1.8</td>
<td>2.0</td>
</tr>
<tr>
<td>Beech wood</td>
<td>0.6</td>
<td>0.8</td>
</tr>
</tbody>
</table>
## Final selection of packing materials

<table>
<thead>
<tr>
<th>Conventional packing material</th>
<th>Alternative packing materials</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Beech Wood</td>
</tr>
<tr>
<td></td>
<td>PES nonwoven</td>
</tr>
<tr>
<td></td>
<td>PES 3D fabric</td>
</tr>
</tbody>
</table>

- ✔ Adhesion of bacteria *C. necator* and biofilm formation
- ✔ Adaptability of the packing material inside the reactor
- ✔ Degradation suffered by the packing materials over time
  - ✔ Accessibility to nutrients by the bacteria
  - ✔ Cost of packing materials

**PENDING:** Biofilm survival over time and CO$_2$ absorption

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Plasma technology

Plasma is a partially ionized gas composed of electrons, ions, photons, atoms and molecules, with negative global electric charge

Surface phenomena:

❑ Surface cleaning without modification of intrinsic properties
❑ Increased fibre surface roughness and surface area
❑ Increased surface energy to promote wetting
❑ Deposition of functional groups onto the surface
❑ Functional nano-coatings deposition (PECVD)

Advantages of plasma technology:

❑ Neither water consumption nor wastewater effluents;
❑ No chemical consumption;
❑ Drying and curing processes are not necessary;
❑ Well-controlled and reproducible technique.

Atmospheric pressure plasma

Low pressure plasma

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Plasma technology

Surface properties for promoting wetting and adhesion of biofilms:
✓ Hydrophobic/hydrophilic
✓ Surface charge
✓ Roughness
✓ Fluid dynamics on surfaces

High surface area for increasing gas and liquid sorption in bioreactors:
✓ Low-cost structured packing
✓ Increasing accumulated attached biomass
✓ Increasing bioreactor performance
Conclusions

• 10 different packing materials have been evaluated by means of surface characterization, behaviour inside the reactor and biofilm adhesion and growth.

• 4 materials have been selected: Raschig rings (standard), PES nonwoven, PES 3D textile and Beechwood chips.

• Plasma treatments will be performed aiming to improve the surface properties of the packing materials and increase the biofilm formation, and thus the 3-HP production.

• The real performance of the packing materials in the TBR will be evaluated during the following months.

• Optimum packing materials with adapted surface properties will be obtained by the end of the project (May 2020).
Thank You

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