# Caffeine removal by Spent Coffee Grounds (SCG) biochar

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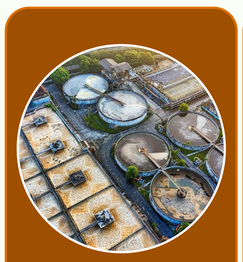
## Research Problem – Scope:

Caffeine is used in beverages, food products and pharmaceuticals

Due to its extensive use, it enters the environment through human activities

Impacts to Ecosystems – Aquatic Life

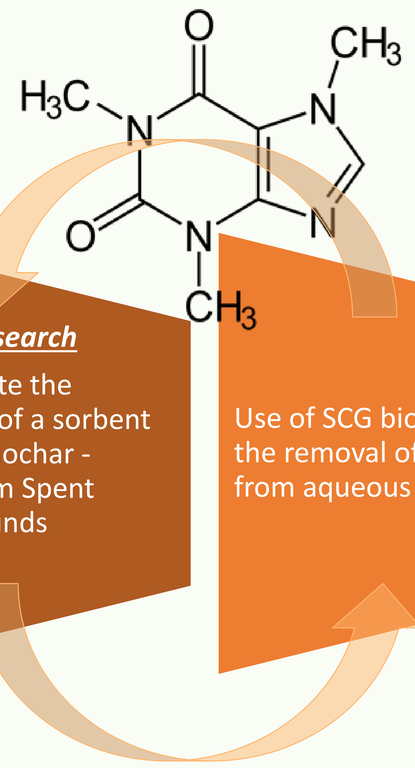


Human pollution marker

Caffeine is considered an **Emerging Pollutant**

Need for removal at source

https://pixabay.com/

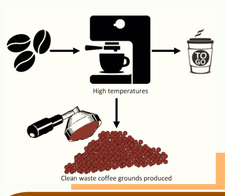


***Scope of research***

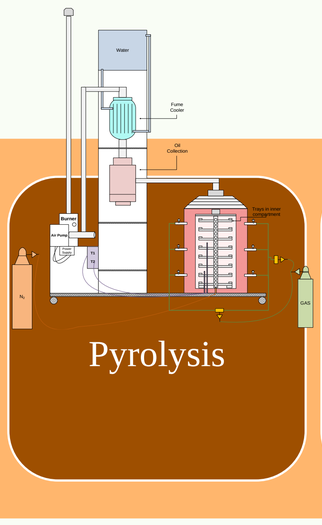
To investigate the production of a sorbent material –biochar - derived from Spent Coffee Grounds

Use of SCG biochar for the removal of Caffeine from aqueous solutions

## Materials and main methods used:




Large quantities of waste produced → Spent Coffee Grounds




Pyrolysis

Biochar Production & Characterization

Sorption of Caffeine

Spent Coffee Grounds (SCG) were collected from the campus cafeteria and were dried at ~ 35 °C.

Samples were subjected to slow pyrolysis in a small scale kiln with capacity of 20–24 kg.
Samples were heated under nitrogen atmosphere for approximately 6–7 °C $min^{-1}$ up to the target temperature (550 °C) and held for 1.5 h.

Produced biochar was characterized through a series of analytical methods such as: SEM, BET, XRD, FTIR analysis.

Adsorption studies were conducted by equilibrating 0.2 - 2 g of SCG biochar with 10 mL of Caffeine solution of 50 ppm concentration (pH = 3.5) in 15 mL glass tubes. The content of the tubes was agitated on a rotator at 125 rpm at constant temperatures for 24 h. After rotation, the suspensions were filtered and the residual caffeine concentration was determined by LC-MS/MS. The amount of caffeine adsorbed at time $t$ and at equilibrium time, $q_t$ (mg/g) and $q_e$ (mg/g), were evaluated using the *Eqs. (1), (2)*, respectively:

Two concentrations of zeolite (clinoptilolite) were also used (0.5 and 1 g) in order to compare its efficiency against the produced biochar. Also, a mixture of biochar/zeolite (0.8:0.2 g) was examined.

$$q_t = \frac{(C_i - C_t)V}{m} \quad (1) \qquad q_e = \frac{(C_i - C_e)V}{m} \quad (2)$$

where $C_i$ (mg/L) is the initial caffeine concentration in the solution, $C_t$ (mg/L) and $C_e$ (mg/L) are the final caffeine concentrations in solution at time $t$ and at equilibrium, respectively. $V$ (L) is the solution volume and $m$ (g) is the dry weight of the adsorbent.

The effect of contact time was explored using a caffeine concentration of 50 mg/L at 25 °C and pH 3.5, following adsorption in successive time intervals between 1 and 480 min.

**LC-MS/MS analysis**: Quantitative analysis of caffeine solutions was performed using an Alliance 2695 Separation Module hyphenated to a Quattro Premier XE triple quadrupole mass spectrometer (Waters Cor. UK). Chromatographic separation was achieved using a Waters Symmetry C18 (2.1 x 150 mm, 3.5 μm) column, kept at 30 °C with $H_2O$ + 0.1 % FA as mobile phase A and ACN + 0.1% FA as mobile phase B. The mobile phase flow rate was 0.3 mL/min and a gradient elution was used. The mass spectrometer was operated in positive ion mode with the following parameters: capillary voltage 3 kV, cone voltage 22 eV, source temperature 120 °C, desolvation temperature 500 °C, desolvation flow 950 L/hr. A single MRM transition was used to detect caffeine (195.1 > 138.0) with a collision voltage of 18 eV.

## Results:

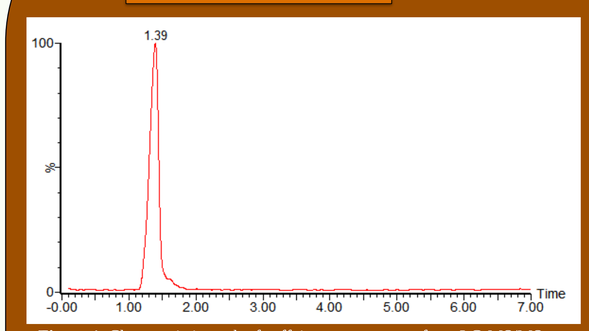



**Figure 1.** Characteristic peak of caffeine measurement from LC-MS/MS

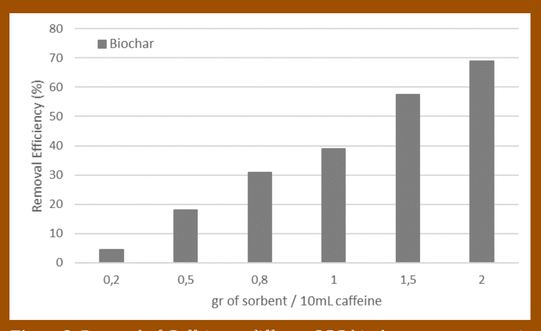



**Figure 3.** Removal of Caffeine vs different SCG biochar mass concentration

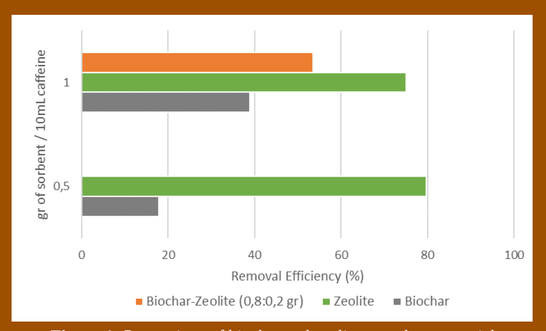



**Figure 4.** Comparison of biochar and zeolite as sorbent materials

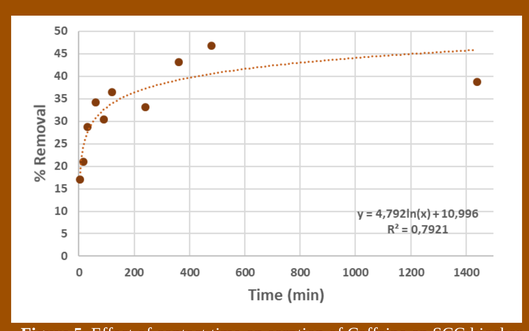



**Figure 5.** Effect of contact time on sorption of Caffeine on SCG biochar

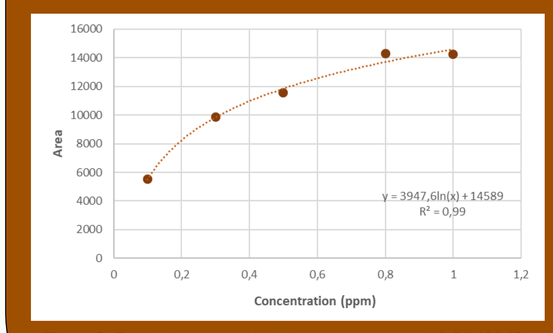
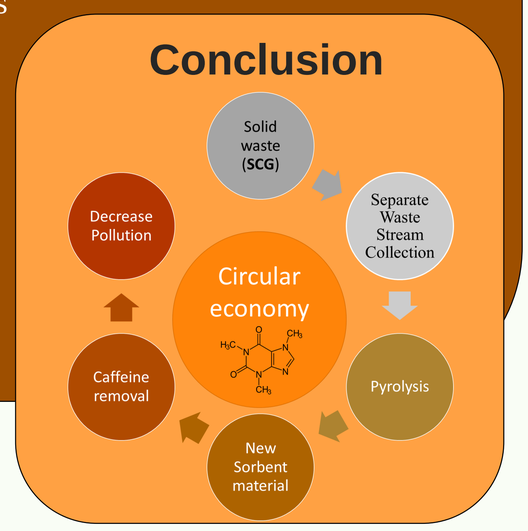



**Figure 2.** Calibration curve of caffeine measurement via LC-MS/MS

- **Figure 1-2** → The method developed achieved to identify and calculate caffeine concentrations up to 1 ppm.
- **Figure 3** → As sorbent mass increases, the % removal of caffeine from solutions is increased.
- **Figure 3** → 70% of caffeine removal was achieved (24 h).
- **Figure 4** → Zeolite has almost twice removal efficiency than biochar at the parameters studied.
- **Figure 4** → The mixture of biochar:zeolite achieved to increase caffeine removal by 28 %.
- **Figure 5** → Short contact times needed (over 60 min) to achieve > 35 % removal efficiencies.
- → Activation of biochar should be examined for increasing the removal efficiency of caffeine.

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