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Keywords: Anaerobic digestion; Acidogenic Fermentation; Hydrothermal Carbonization; Brewery Spent Grain; Winery residues

An integrated process based on hydrothermal carbonization (HTC) and anaerobic treatment of brewery spent grain (BSG) and winery residues (grape skins, GS, and stems, S) was investigated. HTC of BSG was carried out at 180, 210, and 240 °C (1 h), while winery residues were subjected to HTC and co-HTC at 180 °C and 1 h. Furthermore, the generated process waters (PW) were evaluated through acidogenic fermentation (AF) and anaerobic digestion (AD).

For BSG, hydrochar yield remained stable (55–60%) across HTC temperatures, although carbon content and HHV increased up to 210 °C. However, their nitrogen content limits their use as biofuels, although they comply with regulations for soil amendment. All hydrochars from winery residues showed potential as both biofuels and soil amendments, although increasing stem content in co-HTC reduced hydrochar yield, carbon content, and HHV.

Acidogenic fermentation was performed at an SIR of 1.6 and pH 5.5 under mesophilic and thermophilic conditions. The maximum hydrogen yields for BSG's PWs were reached for PW240 with 20.8 mL H₂/gCOD under thermophilic conditions, and for PW180 with 17.5 mL H₂/gCOD under mesophilic conditions. All assays generated similar volatile fatty acids (VFA) concentrations (5–6 gCOD/L). For winery residues, thermophilic AF of PW led to clear inhibition. Under mesophilic conditions, GS and GS:S PWs also showed inhibition, while stem PW obtained a production of 40.2 mL H₂/gCOD. Moreover, final VFAs concentration of stem PW was comparable to those obtained from BSG PWs, but with a higher content of caproic acid, up to 0.8 gCOD/L.

In AD, methane yields from BSG derived PW decreased from 175 to 71 mL CH₄/gCOD as HTC temperature increased from 180 to 240 °C, indicating higher methane potential at lower carbonization temperatures. For winery residues PWs, methane production was similar (230–240 mL CH₄/gCOD), with the highest value reached for stem PW.

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