

Abstract

Closing the Loop in an Efficient Manner—The Feed-Forward Control Solution †

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The Romanian economy is dominated by linear value-chains. Closing the loop in these value chains involves implementing biorefinery processes developed according to pyramid biomass value [1]. The main issue with using the bioeconomy side-streams as feedstocks for sustainable biorefinery processes is their high variability in the recoverable ingredients [2]. The feed-forward control systems adapt the biorefinery technology parameters to the specific features of the processed side-streams [3]. We aimed to develop a feed-forward system integrated into the biorefinery of the cyprinids side-streams, i.e., scale, skin, bones, into high-value products—bioactive peptides and ingredients for 3D- and/or 4D-printed biocompatible scaffolds. The cyprinids side streams were mixed and grounded, and 12 samples from different batches were prepared for feed-forward method calibration. The crude proteins content was determined as $6.25 \times$ nitrogen according to ISO 5983–2/2009. The total collagen content was estimated by determination of the hydroxyproline content—ISO 3496:1994. Two vibrational spectroscopic methods, near-infrared (NIR) spectroscopy and Raman scattering, were used as candidates for the fast and non-destructive feed-forward techniques. The spectra were acquired for grounded sample arranged in plastic trays, with a surface equal to A4 paper (210×297 mm) and a height of 2 cm. The NIR spectra were acquired in the range 900–1700 nm, using a NIRQuest + 1.7 handheld spectrometer (Ocean Insight, Orlando, FL, USA). To acquire the Raman spectra, we used a handheld Raman-HR-TEC 785 nm device (StellarNet, Tampa, FL, USA). Partial least squares (PLS) regression was used to establish correlations between the proteins and collagen content determined by the ISO methods and NIR and Raman spectra. Protease and peptidases were added according to the determined protein/collagen ratio to produce (bioactive) peptides and (printable) polypeptides. To select and validate the spectroscopic feed-forward method, 6 samples were processed according to values estimated by spectroscopic techniques. The yields on peptides and polypeptides were determined by gel electrophoresis. NIR peaks from 1120–1350 nm were proven to have the best correlation with the total protein content— $R^2 = 0.87$. The peaks of Raman spectroscopy correlate well in the range of 850–950 cm^{-1} with the ratio between protein/collagen ratio— $R^2 = 0.78$. Using the ratio between peaks at 861 cm^{-1} and 916 cm^{-1} , a good prediction of the necessary activities of protease and peptidases in the biorefinery process was obtained. Raman spectroscopy at 785 nm is a

functional feed-forward solution and allows for the estimation of fish side-streams, which are specific features for optimal protease/peptidase treatment.

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