FTIR ANALYSIS OF ALGAE POWDER UTILIZED AS NATURAL FILLERS IN 3D PRINTING FILAMENTS

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In this research the algae powder was analysed as potential natural filler for PLA matrix. Namely, algae as an organic material must withstand temperatures ranging from 170 to 230 °C during the short duration of the extrusion and printing processes. The change in colour and odour at these temperatures can be an indicator that material is going through some changes. However, Fourier transform infrared (FTIR) spectroscopy as an efficient, non-destructive analytical technique was used in this study to assess the structural stability of algae powders by comparing functional groups before and after treatment with elevated temperature. By analysing the changes in the spectral signature, it was determined whether thermal exposure causes degradation or whether the material maintains its structural integrity.

MATERIALS AND METHODS

Algae powders used for this research were:

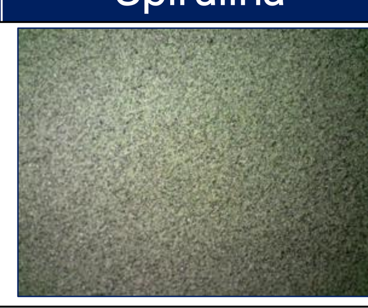
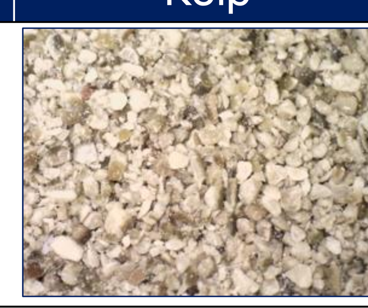
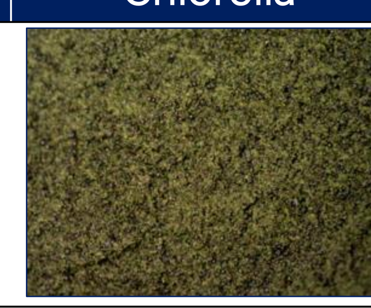
- kelp powder from algae *Ascophyllum nodosum* (Canada),
- chlorella powder from algae *Chlorella spp.* (China),
- spirulina and blue spirulina powder from algae *Spirulina spp.* (China).

All algae powders were:

- subjected into a Nabertherm L9/11 B180 Muffle Furnace to temperature 220 °C for 10 minutes
- captured by Dino-Lite digital microscope before (T_1=25 °C) and after heat treatment (T_2=220 °C)
- recorded on Perkin Elmer Spectrum Two spectrometer equipped with diamond ATR accessory.

RESULTS

Table 1. Photographs of algae before (T_1) and after (T_2) heat treatment

Temperature	Spirulina	Blue spirulina	Kelp	Chlorella
T_1 = 25 °C				
T_2 = 220 °C				

Exposing algae powders to temperatures of 220 °C resulted in the organic material turning black (Table 1). This change of colour in organic algae powder to black at elevated temperatures is primarily due to carbonization.

In Figures 1 and 2 the FTIR spectra of un-treated and in Figures 3 and 4 the spectra of heat-treated algae powders are shown, where significant differences are clearly noticeable between the spectra of samples before (T_1) and after the heat (T_2) treatment.

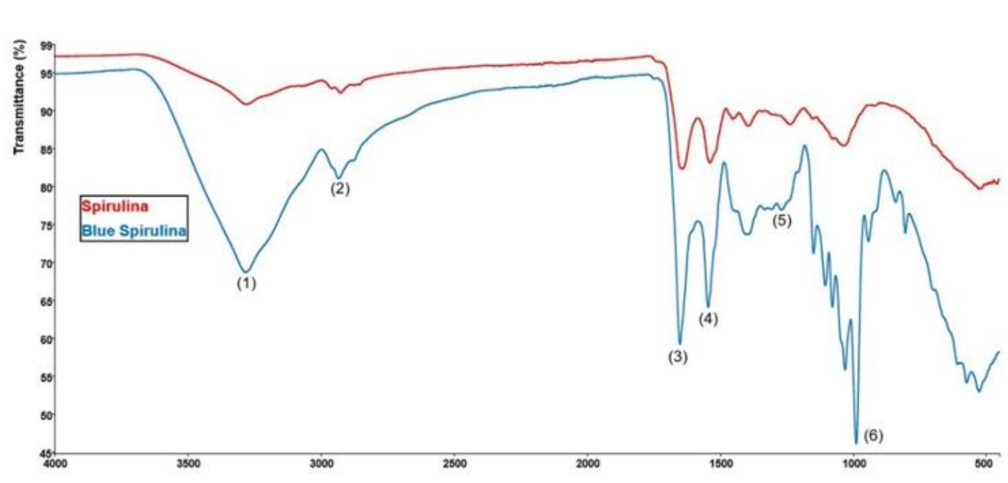


Figure 1. FTIR spectra of un-treated (T_1) spirulina and blue spirulina algae powders

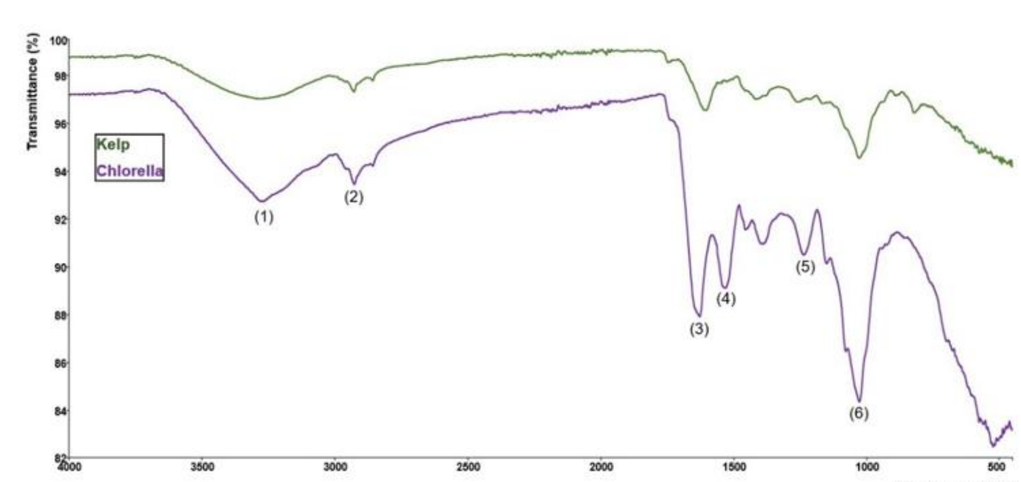


Figure 2. FTIR spectra of un-treated (T_1) kelp and chlorella algae powders

Band 1 (3280-3270 cm^{-1}) - hydrogen bonded O-H stretching vibrations

Band 2 (2933 cm^{-1} or 2925 cm^{-1}) - symmetrical and asymmetrical valence vibrations of the aliphatic groups

Band 3 (1650-1600 cm^{-1}) - asymmetric valence vibrations of the O-C-O carboxylates or C=O stretching in protein Amide I band

Band 4 (1545-1530 cm^{-1}) - Amide II band (proteins)

Band 5 (1545-1530 cm^{-1}) alongside **band 6** (1035-1025 cm^{-1}) - deformation vibrations of the C-H and O-H groups, and to valence vibrations of the C-O bonds

In FTIR spectra of heat-treated spirulina and blue spirulina algae powders (Figure 3) only bands 1, 2 and 4 are still clearly noticeable, and in FTIR spectra of heat-treated kelp and chlorella algae powders (Figure 4), alongside the band 2, noticeable in both spectra, two additional strong bands appear in the kelp spectra.

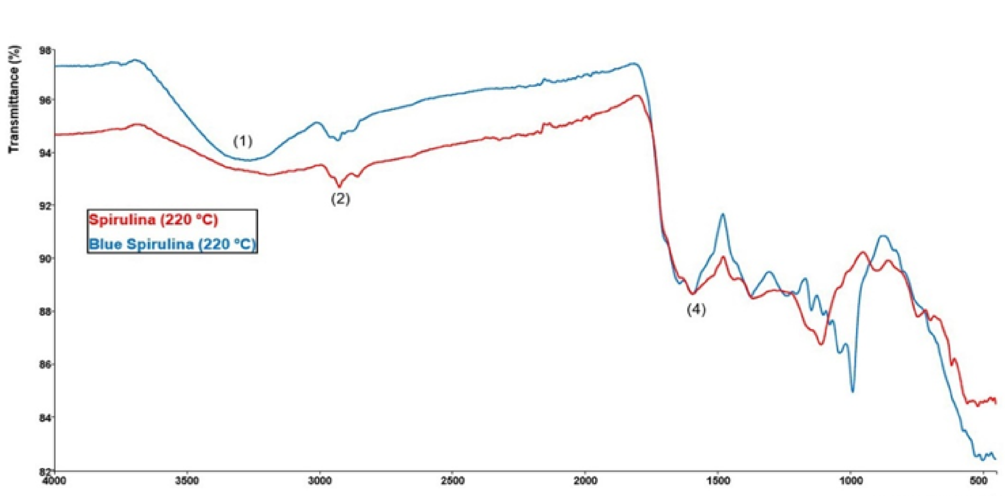


Figure 3. FTIR spectra of treated (T_2) spirulina and blue spirulina algae powders

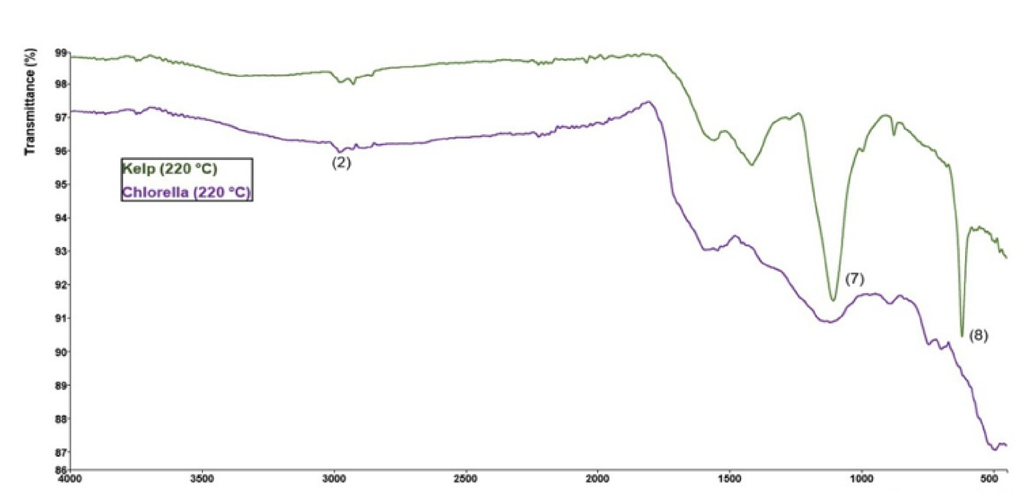


Figure 4. FTIR spectra of treated (T_2) kelp and chlorella algae powders

Band 7 (1105 cm^{-1}) - C-O stretch in cellulose

Band 8 (617 cm^{-1}) - C-H and C-O-S out of plane vibrations

When algae powder is exposed to heat treatment, the material changes. Heat breaks down complex sugars and removes moisture, revealing underlying structures that appear in spectroscopy as entirely new or more pronounced bands in FTIR spectra.

CONCLUSION

FTIR spectrometry is an excellent entry analysis for natural fillers in the production of 3D printing filaments. This analysis helps to identify chemical bonds and thus can show whether the chosen material has chemically broken down during the heat treatment. If the bonds are broken, new peaks for carbonyl groups appear on the spectra, indicating thermal degradation. The FTIR analysis confirmed that a temperature of 220°C will be too high for the PLA-algae filament. Due to organic additives (algae powder), this material is more heat-sensitive and must be printed at lower temperatures.

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