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Physico-Chemical Characterization of Vegetable Oils from Oleaginous Seeds (Croton megalocarpus, Ricinus communis L., and Gossypium hirsutum L.)

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Abstract : According to the Renewable Energy Directive II, the use of palm oil in diesel will be gradually reduced from 2023 and should reach zero in 2030 due to the deforestation caused by its production. Eni aims at finding alternative feedstocks for its biorefineries to eliminate the use of palm oil by 2023. Therefore, the ideal vegetable oils to be used in bio-refineries are those obtainable from plants that grow in marginal lands and with low impact on food-and-feed chain; hence, Eni research is studying the possibility of using oleaginous seeds, such as castor, croton, and cotton, to extract the oils to be exploited as feedstock in bio-refineries. To verify their suitability for the upgrading processes, an analytical protocol for their characterization has been drawn up and applied. The analytical characterizations include a step of water and ashes content determination, elemental analysis (CHNS analysis, X-Ray Fluorescence, Inductively Coupled Plasma - Optical Emission Spectroscopy, ICP– Mass Spectrometry), and total acid number determination. Gas chromatography coupled to flame ionization detector (GC-FID) is used to quantify the lipid content in terms of free fatty acids, mono-, di- and triacylglycerols, and fatty acids composition. Eventually, Nuclear Magnetic Resonance and Fourier Transform-Infrared spectroscopies are exploited with GC-MS and Fourier Transform-Ion Cyclotron Resonance to study the composition of the oils. This work focuses on the GC-FID analysis of the lipid fraction of these oils, as the main constituent and of greatest interest for bio-refinery processes. Specifically, the lipid component of the extracted oil was quantified after sample silanization and transmethylation: silanization allows the elution of high-boiling compounds and is useful for determining the quantity of free acids and glycerides in oils, while transmethylation leads to a mixture of fatty acid esters and glycerol, thus allowing to evaluate the composition of glycerides in terms of Fatty Acids Methyl Esters (FAME). Cotton oil was extracted from cotton oilcake, croton oil was obtained by seeds pressing and seeds and oilcake ASE extraction, while castor oil comes from seed pressing (not performed in Eni laboratories). GC-FID analyses reported that the cotton oil is 90% constituted of triglycerides and about 6% diglycerides, while free fatty acids are about 2%. In terms of FAME, C18 acids make up 70% of the total and linoleic acid is the major constituent. Palmitic acid is present at 17.5%, while the other acids are in low concentration (<1%). Both analyzes show the presence of non-gas chromatographable compounds. Croton oils from seed pressing and extraction mainly contain triglycerides (98%). Concerning FAME, the main component is linoleic acid (approx. 80%). Oilcake croton oil shows higher abundance of diglycerides (6% vs ca 2%) and a lower content of triglycerides (38% vs 98%) compared to the previous oils. Eventually, castor oil is mostly constituted of triacylglycerols (about 69%), followed by diglycerides (about 10%). About 85.2% of total FAME is ricinoleic acid, as a constituent of triricinolein, the most abundant triglyceride of castor oil. Based on the analytical results, these oils represent feedstocks of interest for possible exploitation as advanced biofuels.

Keywords : analytical protocol, biofuels, biorefinery, gas chromatography, vegetable oil
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