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Distillery Residues from *Cistus ladanifer* (Rockrose) as Feedstock for the Production of Added-Value Phenolic Compounds and Hemicellulosic Oligosaccharides

Júnia Alves-Ferreira[1,2,3] · Luís C. Duarte[1] · Ana Lourenço[3] · Luísa B. Roseiro[1] · Maria C. Fernandes[2,4] · Helena Pereira[3] · Florbela Carvalheiro[1]

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Abstract

Cistus ladanifer residues obtained after essential oil distillation were extracted with ethanol and water (CLRext) and subsequently hydrothermally treated (autohydrolysis) in order to selectively hydrolyze hemicelluloses. The extraction removed a significant amount of potentially valuable compounds (40% *w*/*w*, dry basis), foremost, phenolic compounds (0.363 and 0.250 g gallic acid equivalent/g extract, respectively, for water and ethanol). Autohydrolysis was studied under diverse severity factors (log R_o), in the temperature range of 150 to 230 °C. The hydrolyzates mainly contain oligosaccharides, reaching the highest concentration (23.5 g/L) for log R_o of 3.07 (190 °C), corresponding to a yield of 15 g oligosaccharides/100 g dry feedstock. The processed solids are enriched in glucan and lignin. The maximum glucan content (35%) was attained at log R_o of 3.51 (205 °C). Py-GC/MS confirmed the reduction of pentose-derived carbohydrates in the solid after hydrothermal treatment and an increase of syringil units in the lignin compared to the untreated biomass. These results show the potential use of this *C. ladanifer* residue for the production of phenolic extracts, and hemicellulosic oligosaccharides, together with the production of a cellulose- and lignin-rich solid stream.

Keywords Extraction · Fractionation · Hemicelluloses · Hydrothermal treatment · Phenolic compounds · Pre-treatment

Introduction

Rockrose (*Cistus ladanifer*) is one of the most significant natural shrubs in the Mediterranean basin, mainly distributed in countries such as Portugal, Spain, Greece, Italy, Algeria, and Morocco [1, 2]. It is estimated that it extends by an area of ca. 2 million hectares in the south/southwest of the Iberian Peninsula. Specifically, in Portugal, it extends throughout the country, especially in the center and south regions [3, 4]. This plant shows high proliferation, occupying abandoned or unmanaged agricultural areas and may represent a fire hazard in some regions. It is generally recognized that, if not properly controlled, this species is one of the major responsible for fire spreading in the Mediterranean forest ("Montado"), as allegedly, it has some evolutive traits that enable it to survive and rapidly colonize burnt areas [5–7]. Although *C. ladanifer* is being explored industrially [8] as a source of resin (labdanum gum) and essential oils for cosmetic and perfume industries, it is estimated that the global use of *C. ladanifer* is only about 10,000 tons per year, with 80% of the products obtained coming from Spain, mainly from Andalusia [9]. As such, this feedstock can be considered still to be underexplored, especially the biomass remaining after the steam distillation used for essential oil production, which is used only for energy production by combustion, a very

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✉ Florbela Carvalheiro
florbela.carvalheiro@lneg.pt

[1] Unidade de Bioenergia, LNEG - Laboratório Nacional de Energia e Geologia, Estrada do Paço do Lumiar, 22, 1649-038 Lisbon, Portugal

[2] Centro de Biotecnologia Agrícola e Agro-Alimentar do Alentejo (CEBAL)/Instituto Politécnico de Beja (IPBeja), Rua Pedro Soares, s.n. - Campus IPBeja / ESAB, Apartado 6158, 7801-908 Beja, Portugal

[3] Centro de Estudos Florestais, Instituto Superior de Agronomia, Universidade de Lisboa, 1349-017 Lisbon, Portugal

[4] ICAAM - Instituto de Ciências Agrárias e Ambientais Mediterrânicas, Instituto de Investigação e Formação Avançada, Universidade de Évora, Pólo da Mitra, Ap. 94, 7002-554 Évora, Portugal