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Comparative characterization of terpenes from Cola *nitida* (Vent.) Schott & Endl. (Malvaceae) resin using hydrodistillation and headspace (HD vs HS-GC/MS) techniques.

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Cola nitida (Vent.) Schott & Endl.(Malvaceae) is a native tree of tropical West of Africa, popularly known as *gbanja* or *goro,* and is called “noz-de-cola” or “cola” in Brazil. It is popularly used in central Africa for ritual ceremonies and for spiritual protection. Modern analytical techniques revealed that resins are full of antimicrobial compounds bearing different skeletons which are able to inhibit the growth of bacteria and fungus justifying its use in the mummification process[1]. Since the *Cola nitida* (CN) resin has been poorly studied, the work aimed to establish comparatively its chemical profile by using hydrodistillation with gas chromatography coupled to mass spectrometry (HD-GC/MS) or analysis of the resin without solvents through headspace trap for thermal desorption coupled to the HS-GC/MS, in order to evaluate the presence of monoterpenes susceptible to chemical modifications by the HD process[2]. CN resin (2 Kg) collected from the trunk was poured onto DCM/MeOH (1:1, v/v) for 10 days, then filtered and dried *in vacuo* with a rotary evaporator (50ºC, 120 rpm) to obtain 1.8 kg of a colorless gum. A part of this solid (50 g) was hydro-distilled (HD) for 3.0 h using a Clevenger apparatus. The volatile oil was separated from the distillate yielding *ca* 20% of the crude resinous extract. GC/MS analysis were performed on a Perkin Elmer Clarus 680 directly coupled to the mass spectrometer system (Perkin Elmer Clarus SQ8). Elite 5mS non polar fused silica capillary column (30 m x 0.25 mm i.d., film thickness 0.25 µm) was used under the following conditions: oven temperature program from 35ºC (4 min) to 90ºC at 20ºC/min, to 130ºC at 5ºC/min, 130ºC (1 min), and to 250ºC at 15ºC/min; injector temperature 250ºC, carrier gas He, flow rate 1 ml/min; the volume of injected sample was 0.5 µl (25 µg/ml of diluted oil in diethyl ether); split injection technique (10 ml/min); ionization energy 70 eV, in the electronic ionization mode (positive); ion source temperature 180ºC; scan mass range of *m/z* 30-600 Da. and interface line temperature 220ºC. The constituents of essential oils were identified based in comparison with similarity of their mass spectra with those gathered in the NIST-MS library (2010), EI-fragmentation study and fragmentation profile in the reference literature[3]. For the HS-GC/MS analysis, 1 mg of crude resin was prepared with HS Perkin-Elmer 40/110 Trap at 100ºC (10 min), split injection technique (30ml/min) and followed by GC/MS analysis under the same conditions. 26 compounds were identified in HD-GC/MS and 25 compounds in the HS-GC/MS technique and the major compounds were characterized as α-phellandrene and p-cymene in both techniques. The essential oil of CN is composed of only monoterpenes; however, 2 compounds with low molecular weight (2-Tujene and Camphene) were identified exclusively by HS-GC/MS technique and 5 compounds with high molecular weight were identified exclusively by HD-GC/MS thecnique. According to preliminary data, it is

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concluded that the temperature difference of the two techniques can interfere in the characterization of the CN resin.

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