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Method validation using HPLC for the isolation, separation and quantification of some biologically active compounds

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Method validation for the identification, isolation, separation and purification of one typical class of biologically active purine based alkaloid viz. Caffeine was carried out using reverse phase High Performance Liquid Chromatography technique (HPLC) in the isocratic mode. Conventional solvent extraction technique involving differential and incremental solvation procedure by binary solvent mixtures was adopted to extract the total alkaloids present in the samples. Non polar protic constitutive solvent (ethylene glycol), Polar aprotic dissociative solvent (1, 4-dioxane) in aqueous medium at a composition of 20%+70% was used for extraction of purine alkaloids from the samples. UV-visible spectrophotometry was used to ascertain the presence of the alkaloid in the samples. The operational parameters (Caffeine); C-18 column, CH₃CN + MeOH+H₂O at pH =8.8 (phosphate buffer), u =1ml/min, Pinlet= 130-140 bar, λ = 273 nm (VWD), T = ambient at 27°C, η = 10⁻³ N sec m⁻², D = 10⁻⁹ m² sec⁻¹, Φ = 1000, h = 3 and d_p = 5 mm was found to be the optimal parametric combination for the effective analysis of this purine based alkaloid by HPLC. Recovery rate of caffeine from samples are estimated to be: 100%, 97%, 98.5%, 98%, 96% & 98.8% for Lamolate®, Coffee beans, Tea Leaves, soft drink -1 (Pepsi), soft drink -2 (Coke) & soft drink -3 (Thums up) respectively.

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The effect of pre-treatment solutions and time on acetylation of wood flour using vinegar

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Effect of pre-treatments and time on acetylation wood flour obtained from four different wood species grown in Nigeria using vinegar was investigated. Similar weight gains in acetyl were obtained despite varied pre-treatment procedure employed and acetylation of samples increased with increasing time of acetylation. This research work has shown that commercial house hold bleach could be used as substitute for NaOH for fiber pre-treatment and other solvents used for removal of wood extractives. FT-IR spectroscopy was very vital in providing evidence of the success of acetylation as it was used for characterization of important functionalities of acetylated products. Important absorptions in the acetate group were detected and these include; C—O, C=O, —C—CH₃—, and the reduction in —OH absorptions also showed that some level of acetylation occurred. This work further opens door to new application of vinegar in wood industry for surface modification of wood and lignocellulosic fibers for various applications.

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