

SYNTHESIS AND CHARACTERISATION OF GLUCOSE ESTERS OF PALM OIL AND PALM OIL PRODUCTS

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Introduction

Carbohydrate fatty esters have been shown to have good prospect as surfactants (Bobalek, 1977; De Luca et al. 1997; Ducret et al. 1995) but to produce food quality carbohydrate fatty esters in commercial quantities has remained a big challenge. This is because the condensation reaction between the carbohydrates and fatty acids is beset with problems: (i) the two substrates are not mutually soluble; (ii) known mutual soluble solvents are toxic; and (iii) the substrates are rarely stable at the temperatures required for the reaction to occur. In order to circumvent these problems we have adopted a solvent-free interesterification process (Akoh and Swanson, 1990) to produce acetylated glucose fatty esters at relatively low temperatures and in good yield. Structural analysis, e.g. TLC, FT-IR, ¹H- and ¹³C-NMR have shown the products to be mainly mono-substituents. The objective of this project therefore was to find a reaction process that can produce food quality, glucose fatty esters in good yield.

Materials and Methods

Glucose pentaacetate, GPA (99% pure) was purchased from Fluka Biochemica, FAME of PO and PKO, stearic and oleic acids were gifts from Henkel Oleochemical, Malaysia. Buchs Silica gel 60 (particle size 0.063-0.200mm, 70-230 mesh ASTM) and pre-coated silica gel 60 F₂₅₄ TLC plates were obtained from E. Merck (Darmstadt, Germany). All solvents used were of analytical grade and obtained from E. Merck (Darmstadt Germany).

Appropriate fatty acid methyl ester (FAME) was put in a three-necked, round-bottom flask equipped with a magnetic stirrer, stopcocks, vacuum take-off line leading to liquid nitrogen cold-trap and a vacuum pump. The reaction flask was flushed with dry N₂ gas for 30 minutes before admixing the reactants. GPA (5.0mmol) and sodium metal (0.12g) were added and heating commenced with continuous stirring. An oil bath was used to maintain the temperature between 80 and 105°C. The reaction was continued for 4 to 5 hours. The product was neutralised with 1-3ml glacial acetic acid, allowed to cool, dissolved in acetonitrile and decolourised with 2g of activated charcoal. The glucose fatty-acid esters

were purified by separation on a 40x2 cm slurry packed column (packed with silica gel 60, particle size 0.063-0.200mm; 70-230 mesh ASTM). The products were eluted with 10% ethyl acetate in hexane. The ethyl acetate and hexane were evaporated by rotary evaporation to give the products. Glucose esters of palm oil palm kernel oil, stearic and oleic acids were prepared in the same manner.

Results and Discussion

Molecular Structure of product was confirmed from H- and ¹³C-NMR, corroborated with FT-IR spectra. The relative position of the acyl group on the pyranosyl ring was determined from the relative δ ppm shifts of the C=O carbon (168.91-170.98 in ¹³C-NMR), resulting from the shielding effect of the long-chain alkyl group compared to the methyl group in the acetate. For the product, 1-O-acyl-2,3,4,6-tetraacetyl- α -D-glucopyranose, the C=O linked to C₁ gives a δ (ppm) of 0.10 relative to the δ (ppm) of the equivalent in GPA and it is the largest displacement in the class. The interesterification reaction was found to be most effective at temperatures between 80°C and 90°C, 1-20 mHg pressure and mole ration of GPA: FAME 1:3. The yields obtained were between 60-63%. The reduction of surface tension from 72 dynes/cm in de-ionised water to about 38 dynes/cm for these compounds demonstrates their potential as surfactant molecules.

Conclusions

Interseterification provides a convenient route to the production of glucose fatty acid esters without the use of solvents. The reaction products, mono fatty-esters of glucose acetate, are potential surfactant ingredients in food, cosmetics, pharmaceutical and other industries. The acetate group attached to glucose is a good leaving and protective group and, at the same time, enhances the solubility for the glucose moiety in fatty acid methyl ester (FAME). The yield can be further increased through optimisation of this process.

References

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