

SULFOXIDES AND SULFONES AS SOLVENTS FOR THE MANUFACTURE OF ALKYL POLYGLYCOSIDES WITHOUT ADDED CATALYST

Camille Ludot ^{a,b}, Boris Estrine^a, Jean Le Bras ^{*b}, Norbert Hoffmann^b,
Sinisa Marinkovic ^{*a} and Jacques Muzart^b

a Agro-industrie Recherches et Développements, Green Chemistry Department, Route de Bazancourt,

POMACLE, France. Fax: +33 (0)3 26 05 42 89; Tel: +33 (0)3 26 05 42 80; E-mail: s.marinkovic@a-r-d.fr

b Institut de Chimie Moléculaire de Reims, UMR 7312, CNRS-Université de Reims Champagne-Ardenne, UFR des Sciences Exactes et Naturelles, BP 1039, 51687 REIMS Cedex 2, France. Fax: +33 (0)3 26 91 31 66; Tel: +33 (0)3 26 91 32 46; E-mail: jean.lebras@univ-reims.fr

HPLC analysis of organic acids:

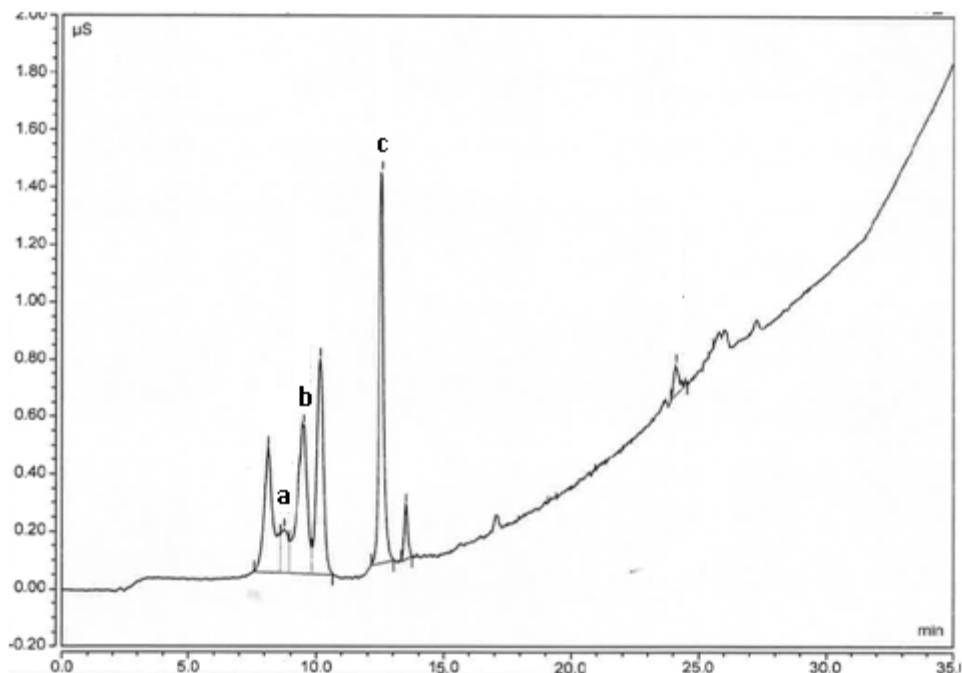
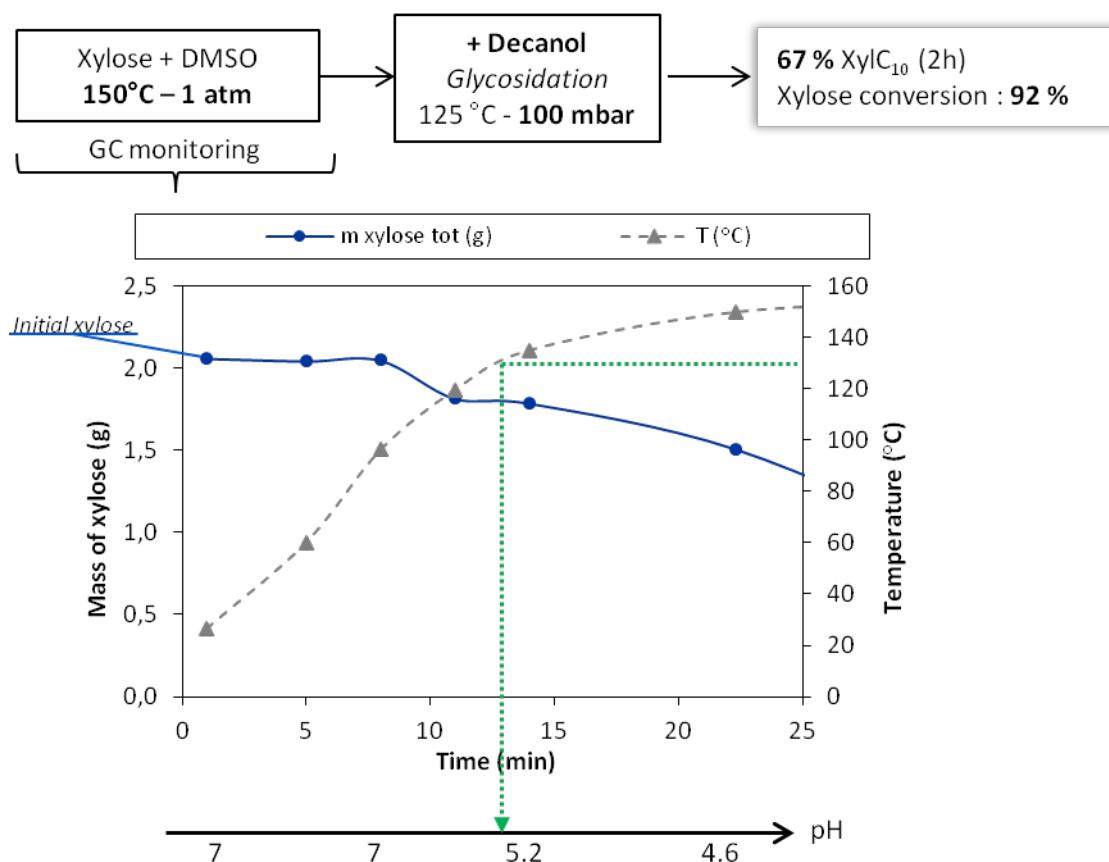


Figure 1. HPLC chromatogram of the thermal degradation of D-xylose in DMSO (a, lactic acid; b, acetic acid; c, formic acid)

Scheme 1: Gas Chromatography monitoring and pH monitoring of the thermal degradation of D-xylose in DMSO



Recycling of DMSO₂:

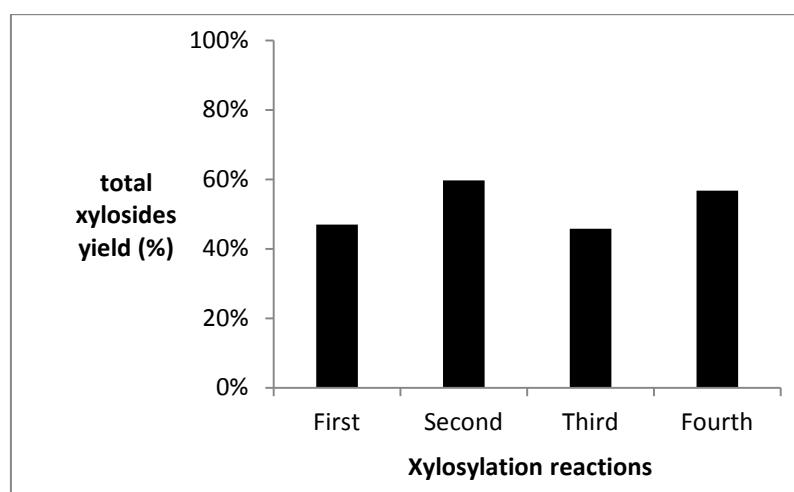


Figure 2. Successive xylosylation reactions in dimethylsulfone. Reaction conditions as described for Table 4

Effects of temperature and added catalyst on glycosidation reactions in sulfolane:

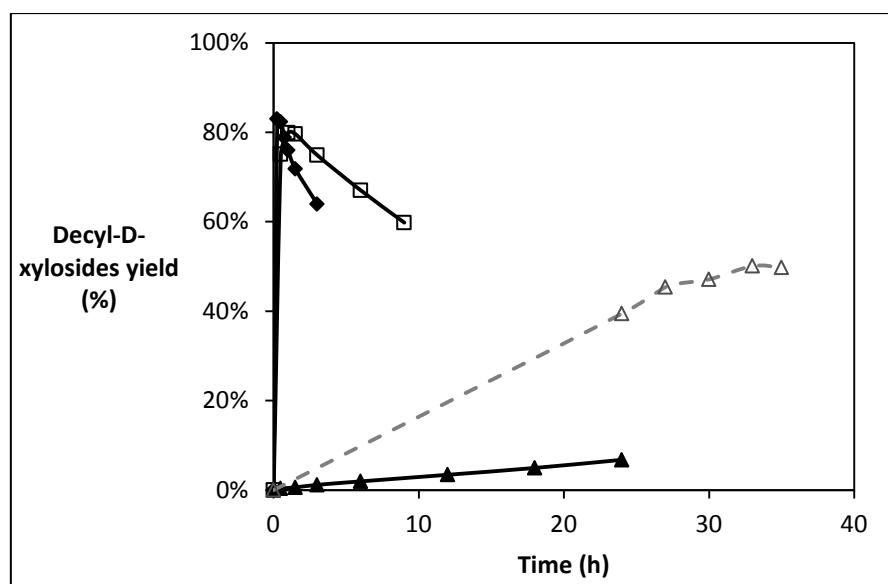


Figure 3. Yield of decyl-D-xylosides as a function of time

T = 150 °C (◆); 125 °C (□); 90 °C (▲); 90 °C in the presence of formic acid as catalyst (△)
Reaction condition: D-xylose (0.033 mol), Sulfolane (6 mol. equivalent based on D-xylose), Decanol (9 mol. equivalent based on D-xylose), air atmosphere

Characterization of decyl-D-xylosides produced by glycosidation of D-xylose with decanol in sulfolane (Table 4, Entry 5):

Biodegradation

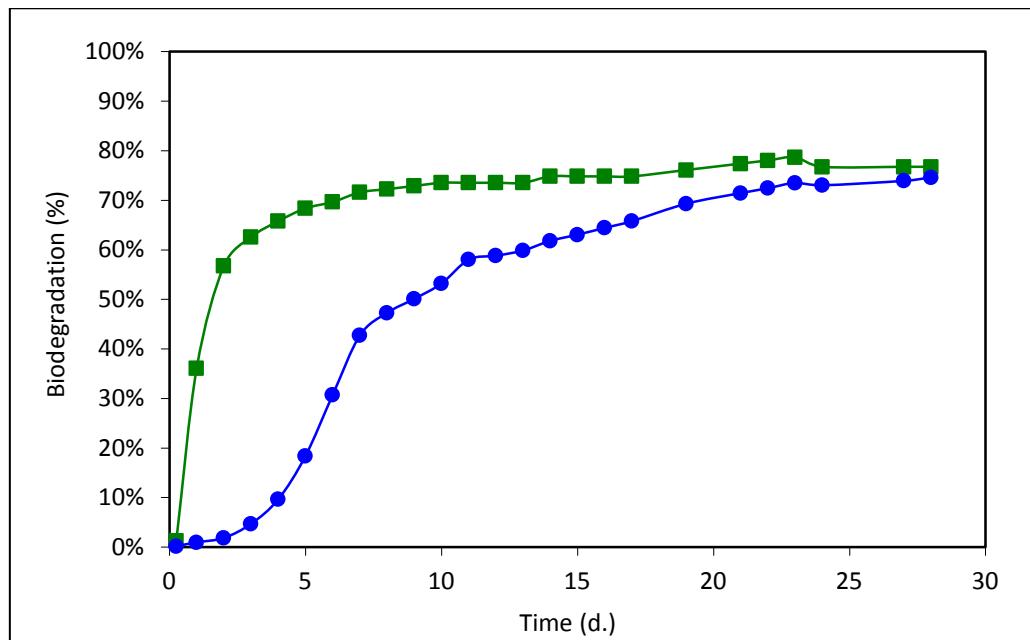


Figure 4. Evolution of the ultimate biodegradability of decyl D-xylosides over 30 days (■, NaOAc (standard); ●, decyl D-xylosides) (according to OCDE 301F standard)

Physico-chemical properties

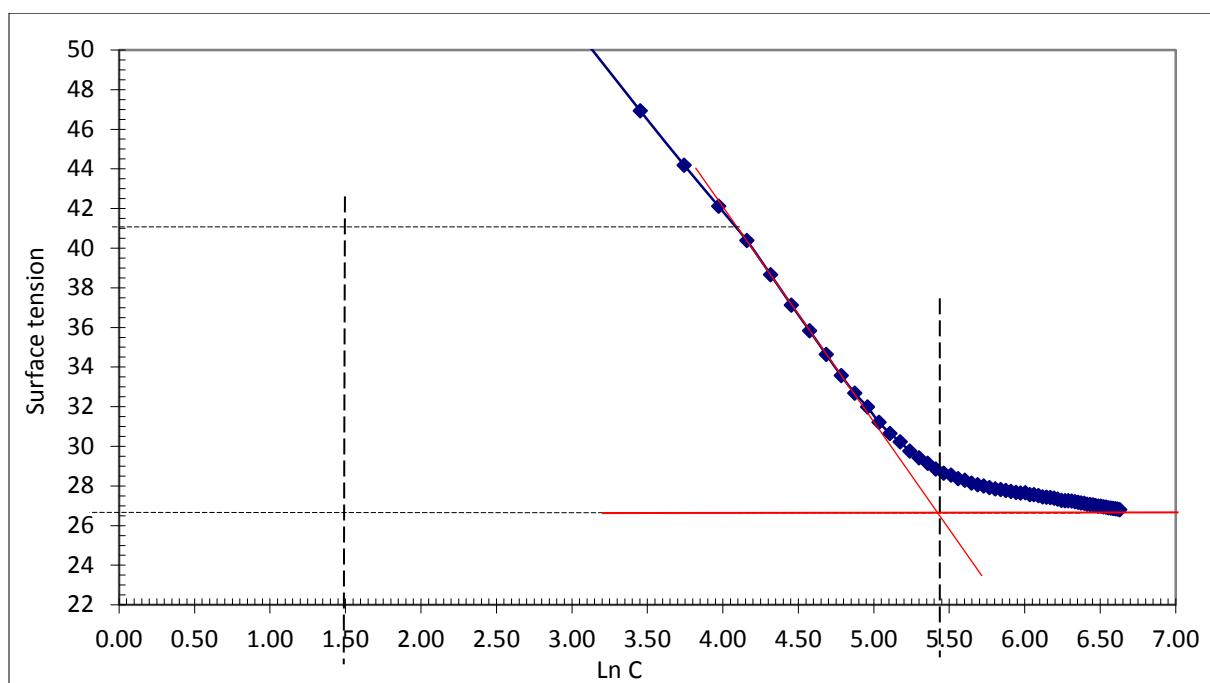


Figure 5. Determination of the surface tension versus decyl D-xylosides concentration¹

¹ S. Marinkovic and B. Estrine, *Green Chem.*, 2010, **12**, 1929

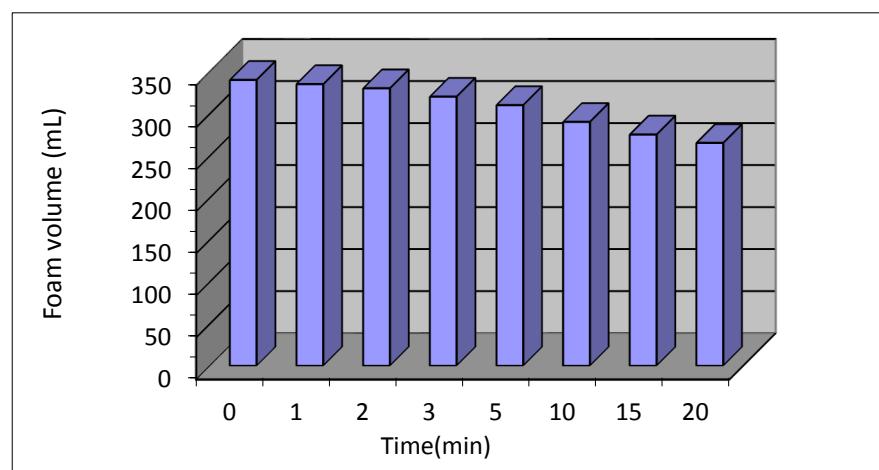


Figure 6.Determination of the foam power of decyl D-xylosides following Ross Miles tests²

Foam production versus time ($50\text{ }^{\circ}\text{C}$) = 340 mL (stability at 20min = 77.9 %)

Wetting power following Draves tests: ($25\text{ }^{\circ}\text{C}$) = $10\text{ s} \pm 1\text{ s}$

² S. Marinkovic and B. Estrine, *Green Chem.*, 2010, **12**, 1929

RMN analysis:

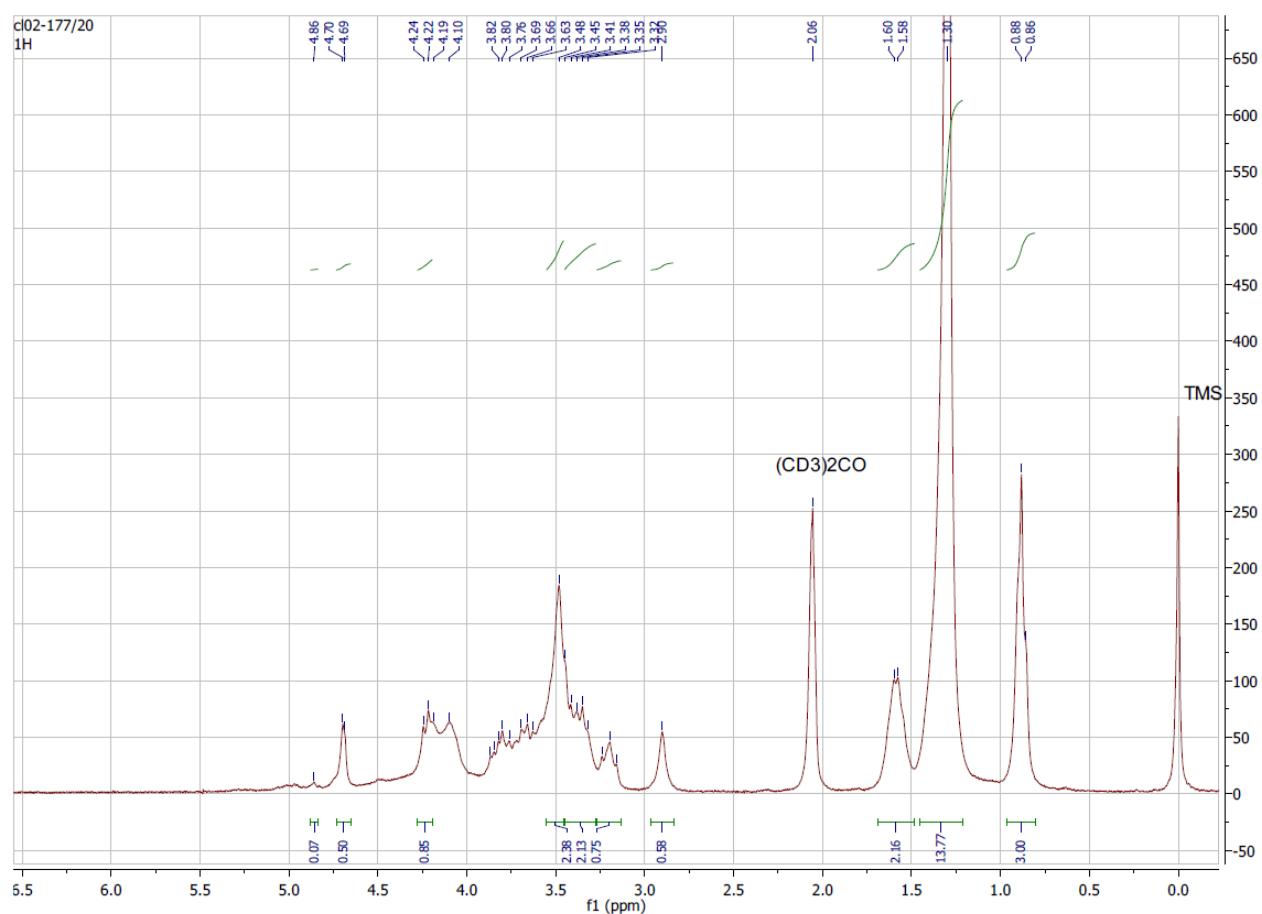


Figure 7. ¹H RMN spectra (solvent $(CD_3)_2CO$)³

³ S. Marinkovic and B. Estrine, *Green Chem.*, 2010, **12**, 1929

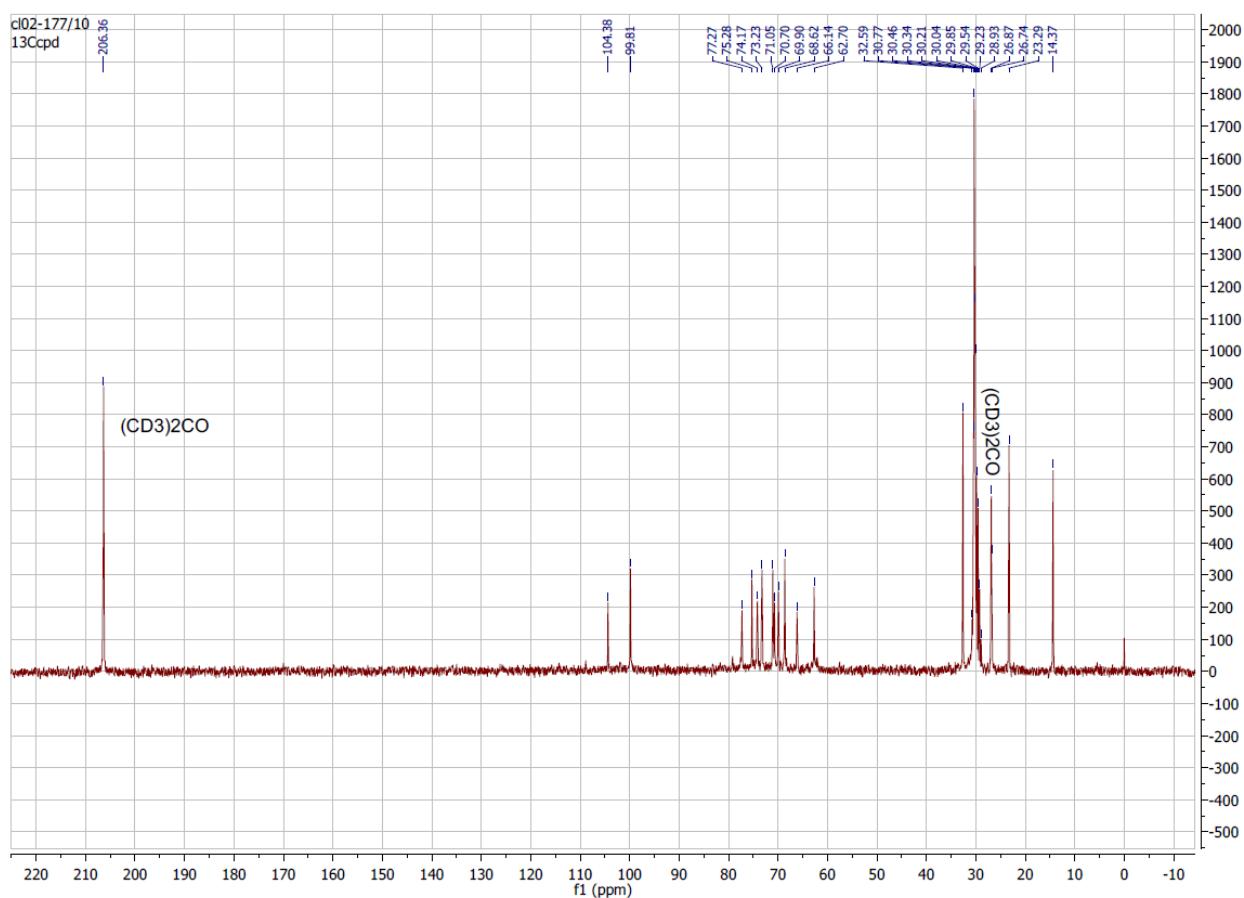


Figure 8. ¹³C RMN spectra (solvent (CD₃)₂CO)⁴

⁴ S. Marinkovic and B. Estrine, *Green Chem.*, 2010, **12**, 1929