Physical and Functional Properties of Some Higher Alkyl Polyglucosides

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Abstract

The higher alkyl polyglucosides are a new class of nonionic surfactants which can be formulated to give properties ranging from soft greases to hard glassy water-soluble solids melting from about 30 C to above 300 C and which are insoluble in common organic solvents. The aqueous solutions do not exhibit inverse solubility with temperature or concentration. They show low eye and skin irritation and a very low degree of toxicity (LD_{50} greater than 35 g/kg). They have a bland taste, are good foamers with low surface tension, are compatible with inorganic detergent builders, and are biodegradable. They show good functionality in various applications such as detergents, food emulsifiers, cosmetic surfactants, pharmaceutical granulating agents and industrial emulsifiers.

Introduction

In 1938, Noller and Rockwell (1) used the Koenigs-Knorr (2) procedure to synthesize *n*-hexyl, *n*-octyl, *n*-nonyl, *n*-decyl and *n*-dodecyl glucosides from sugar. Although they found these glucosides to be surface active, the Koenigs-Knorr technique is a laborious, multistep procedure which is not commercially practical. Since then, others have used inexpensive sugar as the starting material to produce nonionic surface active agents, such as the sucrose esters (3), sucrose ethers (4) and alkyl glucosides (5). A new, feasible, industrial method (13) has been devised for the economical manufacture of higher alkyl polyglucosides.

Higher alkyl polyglucosides are prepared by the new method from straight and branched chain C_{8-18} alcohols and glucose. Crude as well as refined glucose can be used. The products are complicated glucoside mixtures, consisting of alpha and beta, mono, di and poly, glucopyranosides and glucofuranosides. A structural formula for an alpha-alkyl glucopyranoside with 1,4-links can be represented as in Figure 1.

For identification purposes, the alkyl glucosides are specified as containing the average number of glucose units which fit the hydroxyl value found, thus, *n*-decyl-2.5-glucoside contains 2.5 glucose units per terminal *n*-decyl radical.

Experimental Procedures

Properties and Uses of Select Higher Alkyl Glucosides

Appearance. Judged by the naked eye.

Hydroxyl Number. Pyridine base catalyzed method using 2 hr reflux.

Per Cent Free Alcohol. Determined by GLC; 91 × 0.63 cm copper column, 15% carbowax 20 M on chromosorb W, port 330 C, detector 250 C and 180 ma, DMF solvent.

Melting Range. Fisher Johns Apparatus.

Fractionation. Water solution was separated into fractions by increments of acetone. Excess acetone

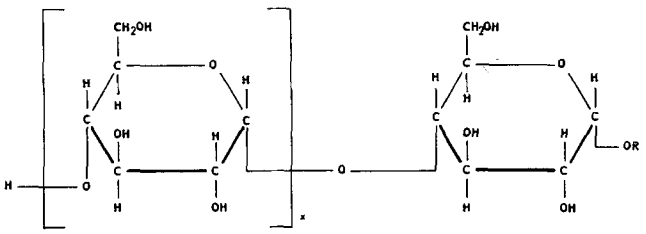

FIG. 1. Structure of a Higher Alkyl Polyglucoside. R is straight or branched chain C_{8-18} Alkyl Radical. X is 1 to 100 units.

was used to precipitate solids from separated lower layer.

Molecular weight. Determined using a Mechrolab Vapor Pressure Osmometer-Model 301, distilled water as solvent and methyl glucoside (Corn Products, Inc.) as the reference material.

Foam Tests. Twenty milliliters of 0.1% w/v of aqueous product contained in 100 ml glass-stoppered graduate was shaken vigorously by hand for 1 min, allowed to stand 1 min and top of foam recorded. Foam tests were also run by the ASTM D-1173-53 procedure.

Critical Micelle Concentration. Obtained by the spectral-dye method using Benzopurpurin 4B. Similar values were obtained from the surface tension concentration curves.

Solubility. One decigram and 2.0 g were placed in test tubes containing 10 ml of the solvent. The mixtures were stirred (where applicable, heated to 75 C) for 1 hr and examined for clarity after standing 24 hr at 25 C. I designates compound insoluble at 1%, PS designates solubility between 1% and 20%, and S designates solubility greater than 20%.

Surface Tension. Measurements were made on a Wilhelmy Tensiometer at 25 ± 0.1 C.

Hydrophile-Lipophile Balance (6). Determined by the optimal emulsion method using Span 80 (HLB 4.3) with glucoside and a naphthenic mineral oil (Sun Golden 91, Sun Oil Co.) of HLB 12.

Viscosity. Run by the ASTM D445-53T procedure. Brookfield viscometer, Model HAT, was also used at different speeds and spindles for comparison.

Humectant Properties. Conducted in standard aluminum cups and results determined by weight (w).

Detergency Tests. Standard tests were run in a Terg-O-tometer (U.S. Testing Co., Inc.) at 50 C, 60 rpm and 1 liter volume using 4 swatches (10 × 10 cm) of American Conditioning House Cotton soiled cloth. Swatches after washing were dried at 82 C for 1 hr and both before washing (original) and after washing swatches were conditioned 16–24 hr at 24 C, 40 RH before reflectance (R) measurements were made on a Hunter Multi-purpose Reflectometer. A 15 min wash followed by two 3 min rinses was used in hard water of 60:40-Ca/Mg. Relative increase in reflectance was calculated by

TABLE I

Properties of Some Higher Alkyl Polyglucosides

Alkyl radical	Glucose units	Appearance	Analyses		Melting range C
			Hydroxyl No.	Per cent free alcohol	
n-Decyl	1.3	Soft amber wax	716	0.5	70– 72
n-Decyl	2.3	Glassy amber wax	802	2.7	95–100
n-Decyl	4.0	Light tan powder	900	0.3	
Oxo-decyl	2.2	Glassy amber solid	820	0.9	108–110
Oxo-decyl	4.0	Light tan powder	907	0.1	195–200
n-Dodecyl	2.5	Hard amber wax	786		
Oxo-tridecyl	1.5	Soft amber wax	651	1.6	60– 70
Oxo-tridecyl	4.0	Light cream powder	859	0.6	217–219
Oxo-tridecyl	9.0	Light tan powder	943	0.9	290–300
n-Tetradecyl	2.0	Hard amber wax	894	0.8	110–120
n-Hexadecyl	1.5	Light tan powder	629	6.0	104–106
n-Octadecyl	1.3	Hard brown wax	525		50– 60
n-Octadecyl	7.0	Light tan powder	872	0.2	>300
n-Oleyl	1.0	Soft amber grease	459		30– 40
n-Oleyl	5.0	Light tan powder	831	1.5	190–200

TABLE II

Fractionation of *n*-Decyl-4.0-Glucoside by Water-Acetone Mixture

Fraction No.	Per cent fraction, wt	Per cent free alcohol	Hydroxyl No.	Calculated glucose units from[a]		Foam height, ml (graduate)	CMC[b] g/dl
				Hydroxyl No.	Molecular wt		
Crude			893	4.0	10.5	53	0.107
1	0.5		769	1.6		94	
2	4.8	0.6	774	1.6	4.3	95	0.080
3	5.3		871	3.0		90	
4	5.8	0.5	897	4.0	6.7	80	0.108
5	5.6		904	4.0		73	
6	13.6	0.02	896	4.0		58	0.11
7	8.8		883	3.0		50	
8	8.3		838	2.0		47	
9	11.9		867	3.0		44	
10	7.8	0.7	856	3.0	6.7	45	0.101
11	11.4		856	3.0		42	
12	11.4		849	3.0		40	
13	4.8	0.8	792	2.0	21.0	40	

[a] From material balance 9.0.
[b] Critical micelle concentration.

TABLE III

Foam Test—ASTM D-1173-53

Alkyl radical	Glucose units	Foam height in CM at 50 C							
		0 ppm Water				300 ppm Water			
		0.1% Conc.		1% Conc.		0.1% Conc.		1% Conc.	
		0 Min	5 Min	0 Min	5 Min	0 Min	5 Min	0 Min	5 Min
n-Decyl	1.3	20.0	18.5	23.5	23.0	5.5	5.5	24.0	23.5
n-Decyl	1.8	17.0	16.5	24.5	24.5	10.0	10.0	24.5	24.0
n-Decyl	2.3	14.5	14.5	24.0	23.5	12.0	11.5	25.0	24.5
Oxo-decyl	2.0	6.0	6.0	26.0	25.0	4.0	2.0	21.5	20.0
Oxo-decyl	4.6	13.1	11.0	18.5	18.5				
Oxo-tridecyl	4.0	11.8	11.2	14.0	14.0				
Oxo-tridecyl	8.0	12.5	12.0	15.5	15.5				
n-Octadecyl	5.0	12.0	11.1	13.5	13.5				
8 POE-Lorol[a]		9.5	9.0	17.6	16.0	9.0	8.5	19.0	18.0

[a] Polyoxyethylene lauryl alcohol.

TABLE IV

Solubility at 25 C[a]

Alkyl radical	Glucose units	Water	Ethylene glycol	Glycerol	C Cl_4/ perclene	Ethyl alcohol/ isopropyl alcohol	Acetone/ ethyl ether/ esters/ dioxane	Vegetable and mineral oils	Benzene[b]/ mineral spirits
n-Decyl	1.3	S	PS	PS	I[c]	I[d]	I	I	I
n-Decyl	2.3	S	S	S	I	I	I	I	I
n-Decyl	4.0	S	S	PS	I	I	I	I	I
Oxo-decyl	2.2	S	PS	I	I	I	I	I	I
Oxo-decyl	4.0	S	PS	PS	I	I	I	I	I
Oxo-tridecyl	1.5	PS	PS	I	S	I	I	I	I
Oxo-tridecyl	4.0	S	S	I	I	I	I	I	I
Oxo-tridecyl	9.0	S	PS	I	I	I	I	I	I
n-Tetradecyl	2.0	I	PS	I	I	I	I	I	I
n-Hexadecyl	1.5	I	PS	I	I	I	I	I	I
n-Hexadecyl	5.0	S	PS	PS	I	I	I	I	I
n-Octadecyl	1.3	I	I	I	I	I	I	I	I
n-Octadecyl	7.0	S	PS	S	I	I	I	I	I
n-Oleyl	1.0	I	I	I	S	I	I	I[e]	I[f]
n-Oleyl	5.0	PS	PS	S	I	I	I	I	I

[a] Solubility: I, <1% soluble; PS, 1–20% soluble; S, >20% soluble.
[b] All glucosides soluble (S) in pyridine.
[c] PS in C Cl_4.
[d] S in ethyl alcohol.
[e] S in mineral oil.
[f] S in benzene.

TABLE V

Surface Tension (dynes/cm) and HLB

Alkyl radical	Glucose units	1.0% Conc.	0.01% Conc.	HLB[b]
n-Decyl	1.8	25.2	46.4	
n-Decyl	2.3	27.7	49.0	17.8
n-Decyl	4.0	35.7	64.5	
Oxo-decyl	2.0	26.8	50.8	15.8
Oxo-decyl	4.0	31.2	63.7	
Oxo-tridecyl	1.5	27.0	26.9	14.6
Oxo-tridecyl	4.0	29.1	52.5	12.8
Oxo-tridecyl	9.0	26.1	52.0	
n-Hexadecyl	5.0	38.9	49.3	
n-Octadecyl	7.0	45.5	49.6	11.4
n-Oleyl	5.0	31.6	39.1	
ABS[a]		37.6	46.2	

[a] Linear Ultrawet K, Atlantic Refining Co.
[b] Hydrophile-lipophile balance.

setting the standard at 100% and computing from:

Units of soil removed = washed R − original R.

Biodegradability. Conducted according to Soap and Detergent Association Determination of ABS/LAS Biodegradability-Shake Culture Test using a Gyrotory Shaker (New Brunswick Scientific Co.) at 22 C, 55 RH.

Dry Cleaning. Standard tests were run using a Terg-O-tometer (U.S. Testing Co., Inc.), 400 ml Perclene Solvent (duPont), 30 min wash, 60 rpm, 21–24 C, no rinse and 2 soiled, 2 unsoiled (10 × 10 cm) swatches of viscose rayon (Testfabrics, Inc.). Swatches were dried at room temperature 4 hr, conditioned overnight at 24 C, 65 RH and their reflectance (R) determined on a Hunter Reflectometer using a green filter.

Units of soil removed = washed R − original R × 100.
Units of soil deposited = original R − washed R × 100.

Water Solubilization. Ten millilter increments of water (0.1, 0.2, 0.3, 0.4 and 0.5 ml) were added to each 100 ml of 1% (w/v) agent in Perclene and shaken 10 min for each 0.1 ml increment (30 min for 0.3 ml) on an Atlab Shaker (A. H. Thomas Co.). Solubility noted after shaking and after one week standing at room temperature.

Antistatic Activity Test. Determined on the Atlab Static Tester which rubs (10 cm stroke, 80 cycle min) a 2.5 × 30.5 cm strip of fabric (skein or yarn) for 1 min across a pair of static-generating rods (Teflon) and a single stainless steel rod collects any charge which is measured on an electrostatic voltmeter. Scoured spun Dacron strips (Testfabrics, Inc.) containing 1% padded, on agent (dry w fabric) were dried and conditioned 16 hr at 24 C, 40 RH before measurement.

Textile Lubricant. Run on Atlab Friction Tester FT-2 (Custom Scientific Instruments, Inc., Kearny, N.J.) with 15 g initial tension, 1080° twist and 1 cm/min speed for yarn to yarn (Y/Y) tension and 15 g initial tension, 180° twist, 1,000 cm/min speed over stainless steel pin for yarn to metal (Y/M) tension. Lubricant (1% dry w yarn) was applied using the Atlab Finish Applicator (Precision Machine & Development Corp., New Castle, Del.) and the yarn conditioned 16 hr at 24 C, 40 RH before measurement of grams tension.

Softener. Determined on four fabric samples (18 × 45 cm), three containing 1% padded-on agent (dry w fabric), one as control (distilled water by a three-member softness evaluation panel. The four folded, unidentified samples are randomly judged three separate times by each panelist and the 12 ratings averaged with 1 assigned to the softest rating and 5 to the harshest.

Granulating Agent. Two per cent and 5% (w/v) aqueous solutions (50 ml) used to granulate 250 g of No. 40 mesh mannitol in upright Hobart mixer. After drying at 52 C, the granules are screened for analyses and also mixed with 3% magnesium stearate (Patterson-Kelly twin shell blender) for formation of tablets on a Stokes B-2 rotary machine equipped with four bevel edge, flat face, single score punches (12.7 × 4.7 mm). Hardness measured by a Strong-Cobb Arner hand-operated hardness tester.

Tablet Lubricant. Three per cent dry agent (90–100 mesh) was mixed with mannitol and the blend used in preparing tablets (see Granulating Agent). Flow of blend in machine, binding of dies, and shape of tablets were noted.

Ice Cream. Standard procedure using butter fat (40% cream) 12%, serum solids 11%, sugar 15%, stabilizer (CMC) 0.2%, emulsifier 0.1%, and remainder per cent w water. During freezing, samples are examined every 2 min for overrun (calibrated stainless steel 234 ml density cup) and dryness (gloss using Gardner Laboratory Portable Glossmeter). After freezing, the extruded ribbon is

TABLE VI

Viscosity in Centipoise of Aqueous Solutions at 22 C

Per cent wt	*n*-Decyl-1.8 glucoside	Oxo-tridecyl-6-glucoside (ASTM)	Oxo-tridecyl-6-glucoside (Brookfield)
0	0.986	0.986	
5	1.49	1.04	
10	2.75	1.43	
30	26.10	.....	
40	98.7	12.8	40–45
50	366.	35.	67–93
60	1,250.	146.	202–248
70	1,480.	1,170.	1760–1850
Nujol[a]	218.		160–230

[a] Extra heavy petroleum oil, Plough, Inc.

TABLE VIII

Biodegradability by SDA Shake Culture Test

Alkyl radical	Glucose units	Foam loss %, Day 3	Surface tension dynes/cm² Day 3	Day 7	Day 8
n-Decyl	2.3	100	70	70	70
Oxo-decyl	2.9	100	64	66	67
LAS[a]		100	70	71	71

[a] Soap and Detergent Association's (SDA) linear alkyl sulfonate.

TABLE VII

Humectant Properties

Alkyl radical	Glucose units	Per cent wt loss; 50% w/w aqueous solution: 40% RH; 24 C, 40 min	40% RH; 24 C, 80 min	40% RH; 24 C, 120 min	75% RH; 30 C, 40 min	75% RH; 30 C, 80 min	75% RH; 30 C, 120 min	Per cent gain; solid 75% RH; 30 C, 4 days
n-Decyl	1.8	17.3	30.4	38.9	10.7	12.6	12.7	7.8
n-Decyl	2.3	15.5	27.6	36.2	21.2	18.5	17.6	9.8
Oxo-tridecyl	6.0	8.1	9.6	11.7	4.0	5.0	4.9	13.1
n-Octadecyl	9.0	5.8	10.0	14.7	1.3	1.6	0.8	11.3
Sorbitol		1.2	3.4	5.0	2.5	3.0	3.1	28.0
Glycerine		1.2	1.5	2.1	1.0	0.7	0.5	

TABLE IX

Toxicological Properties[a]

Alkyl radical	Glucose units	Primary irritation to rabbit skin[b]		Irritation to mucosa of the albino rabbit eye[b]			
		Irritation Index	Classification	2 Sec wash	Classification		No. positive eyes/ No. tested
					Kay and Calandra	Fed. Reg.	
n-Decyl	2.3	0	Nonirritant	No	Moderately	Pos.	4/6
n-Decyl				Yes	Irritating		0/3
Oxo-decyl	2.9	0	Nonirritant	No	Moderately	Pos.	4/6
Oxo-decyl				Yes	Irritating		0/3
Oxo-tridecyl	4.0	0	Nonirritant	No	Mildly	Neg.	1/6
Oxo-tridecyl				Yes	Irritating		0/3
n-Octadecyl	6.0	0	Nonirritant	No	Non-	Neg.	0/6
n-Octadecyl				Yes	Irritating		0/3

[a] Acute oral toxicity: LD_{50} of *n*-octadecyl-9.0-glucoside is >35.5 g/kg. No deaths at this high level.
[b] Fifty per cent conc. w/v.

TABLE X

Detergent Effectiveness of Heavy Duty Laundry Detergents

Alkyl radical	Glucose units	Relative increase in reflectance		
		Solid formula[a]		Liquid formula[b]
		0 ppm Hard H_2O	300 ppm Hard H_2O	300 ppm Hard H_2O
n-Decyl	1.4	96	89	120
n-Decyl	2.3	100	95	
Oxo-decyl	2.0	.....	86	
Oxo-decyl	2.9	.....	70	
Oxo-tridecyl	2.2	99	82	
Oxo-tridecyl	5.0	100	60	
n-Hexadecyl	1.5	100	62	
n-Hexadecyl	5.0	101	81	134
n-Octadecyl	1.3	96	58	
n-Octadecyl	8.0	100	80	130
Renex 690[c]			95	
Ivory snow[d]			77	
Ultrawet K[e]		100	100	
Wisk[f]				100

[a] Fifteen per cent detergent; 35% STPP; 10% Metso; 39% Na_2CO_3; 1% NaCMC; 0.25% conc. (Metso is Na metasilicate, Philadelphia Quartz Co.)
[b] Fifteen per cent detergent; 35% TKPP; 49% distilled water; 1% NaCMC; 0.2% conc.
[c] Ten POE nonylphenol; Atlas Chemical Industries, Inc.
[d] Procter and Gamble Co., Na soap.
[e] Atlantic Refining Co., linear alkyl aryl sulfonate.
[f] Lever Brothers Co., formulated liquid laundry detergent.

TABLE XI

Compatibility in Liquid Detergent

Alkyl radical	Glucose units	Per cent by weight[a]			Appearance, one week
		TKPP	H_2O	CMC	
n-Decyl	2.3	20	65		Clear, liquid
n-Decyl	2.3	40	45		Clear, liquid
n-Decyl	2.3	50	35		Clear, liquid
n-Decyl	4.0	50	35		Clear, liquid
Oxo-tridecyl	8.0	50	35		Clear, liquid
n-Hexadecyl	5.0	50	35		Clear, liquid
n-Octadecyl	8.0	50	35		Clear, liquid
n-Decyl	2.3	20	64	1	Cloudy, some separation (CMC)
n-Decyl	2.3	35	49	1	Haze, some separation (CMC)
n-Decyl	2.3	50	34	1	Thick paste
n-Decyl	4.0	35	49	1	Gel separation (CMC)
n-Hexadecyl	5.0	35	49	1	Clear
n-Octadecyl	8.0	35	49	1	Partial separation (CMC)
WISK[b]					Cloudy, some separation, solid

[a] Fifteen per cent glucoside in all formulas.
[b] Formulated liquid laundry detergent, Lever Brothers Co.

TABLE XII

Dry Cleaning of Rayon, 1% W/V Conc.

Alkyl radical	Glucose units	Water sol.[b]	Units of soil		Static volts (Dacron)
			Removed	Deposited	
Oxo-tridecyl	1.5	0.15	8.5	5.0	
Oxo-tridecyl	2.0	0.20	11.3	3.8	>2000
n-Oleyl	1.0	0.30	11.6	16.1	>2000
50% *n*-Oleyl 50% G-711		0.10	13.1	13.9	150
G-711[a]		0.05	10.4	3.7	0

[a] Amine salt of ABS, Atlas Chemical Industries, Inc.
[b] Water solubilization, gram water soluble in 100 ml.

TABLE XIII

Yarn Lubricant and Textile Softener

Alkyl radical	Glucose units	Lubricant[a]				Cotton[a] softener
		Dacron		Nylon		
		Yarn/ yarn	Yarn/ metal	Yarn/ yarn	Yarn/ metal	
n-Decyl	1.3	62 ± 0	344	44 ± 0	200	4
Oxo-decyl	2.2	158 ± 0	98	216 ± 0	44	5
Oxo-tridecyl	2.0	132 ± 0	120	180 ± 0	48	5
n-Octadecyl	9.0	66 ± 6	86	63 ± 11	16	5
G-263[b]		28 ± 10	86	31 ± 8	72	
Distilled water		44	100			5

[a] One per cent by weight of dry material.
[b] Atlas Chemical Industries, Inc., cetyl ethyl morpholinium ethosulfate.

TABLE XIV

Tablet Granulating Agent and Lubricant

Alkyl radical	Glucose units	Per cent conc.	Sieve analysis, % wt. on sieve							Granule[a] hardness	Tablet[b] wt	Lubricant[c]
			No. 12	No. 16	No. 20	No. 40	No. 60	No. 80	Pan			
n-Decyl	1.8	2	0.2	11.8	16.9	18.8	10.6	6.0	35.5	Soft	425	
n-Decyl		5	....	3.3	16.9	21.6	11.1	7.1	39.5	Soft	410	
n-Decyl	2.3	2	....	2.8	18.5	20.7	10.9	6.4	40.7	Soft	400	
n-Decyl		5	....	3.4	20.7	25.1	10.8	5.8	37.9	Soft	410	
Oxo-tridecyl	6.0	2	....	7.1	25.1	21.5	9.3	4.8	32.4	Soft	420	Binds
Oxo-tridecyl		5	....	10.5	28.4	21.4	8.3	4.3	27.5	Soft	400	
n-Octadecyl	9.0	2	....	14.3	32.6	26.1	9.8	4.2	12.9	Hard	440	Binds
		5	0.1	13.0	31.0	25.6	10.6	4.9	15.2	Hard	435	
n-Octadecyl Mg stearate	1.2											Sl. bind No bind

[a] All granules demonstrated good flow.
[b] All tablets had a good appearance.
[c] Three per cent on mannitol.

TABLE XV

Food Emulsifier

Glucoside		Ice cream, 0.1% conc.				Whipped topping 1.0% conc.						Foam mat drying	
		Max. overrun		Body[b]	Ribbon	Max. overrun			Body and peak	Funnel			
		Per cent[a]	Dryness			Per cent	Time, min	Dryness		Curd	ml	Foam[c] g/ml	Peak
n-Decyl,	4.0	60	6	Fair	Good	80	8	5	Good	Sl.	8	**0.54**	**Fair**
n-Hexadecyl,	5.0	73	7	Good	Fair	227	4	32	None[d]	No.	3	0.55	Fair
n-Octadecyl,	8.0	72	8	Good	Good	267	4	32	None[d]	Sl.	4	0.57	Fair
n-Decyl,	4.0[e]	76	8	Poor	Poor								
n-Hexadecyl,	5.0[e]	73	8	Good	Good								
n-Octadecyl,	8.0[e]	74	11	Sl. Thin	Sl. Thin								
Control		64	8	Good	Good	267	4	13	Poor	Yes	20	Did not aerate	
Tween Mos 100VS[f]		82	7	Good	Good								
Atmos 150VS[g]						175	14	11	Good	No	1		

[a] Time maximum overrun 15–18 min.
[b] All had a slightly grainy texture.
[c] Whipping time 30 min. at 0.35% concentration.
[d] Foamy.
[e] Ratio by weight 80:20, Atmul 84/glucoside. Atmul 84 is mono- and diglycerides from edible fats, Atlas Chem. Ind., Inc.
[f] Atlas Chemical Industries, Inc., mixture of mono- and diglycerides and Tween 80 (20 POE Sorbitan monooleate).
[g] Atlas Chemical Industries, Inc., mono- and diglycerides from vegetable source.

examined visually for body, texture and ribbon formation.

$$\text{Per cent overrun} = \frac{\text{unit mix w} - \text{ice cream w}}{\text{ice cream w}} \times 100$$

Whipped Topping. Standard procedure using fat 34%, sodium caseinate 3%, sugar 8%, HV-CMC 0.36%, Carrageenin 0.04%, disodium phosphate 0.10%, emulsifier 1%, and remainder per cent w water. Examine for body and peak. Sample placed in standard 45° glass funnel and after 24 hr, noted for texture and ml of water seeping through funnel into graduate.

Foam Mat Drying. An amount of 0.35% w was added to tomato paste and whipped in a 3 qt. Hobart bowl at speed 2 with wire whip.

Bread. Standard procedure using sponge formula of (w flour as 100) flour 65%, water 37.2%, yeast 2.5%, yeast food 0.5% and dough formula of flour 35%, sugar 8%, salt 2%, lard 3%, non-fat dry milk solids 6%, water 28.2%, emulsifier 0.25–0.5%. The volume (cc) of loaf was measured (loaf volumeter-AACC) 1 hr after baking and wrapped in Cellophane (No. 300 MSD 53), sealed and stored at 24 C, 50 RH for six days. After three and six days, loaves were sliced (½ in. Oliver Slicing Machine No. 777), and the middle five slices used to determine softness on the Instron model TM, standard Speed, and also observed for crumb, grain, texture, crust and color. Each result is an average of 12 loaves.

Bakers Yellow Cake. Standard procedure, using (w flour as 100) powdered egg yolk 20%, cake flour 100%, sugar 130%, non-fat dry milk solids 12.5%, salt 1.87%, baking powder 5%, emulsified shortening 40.9%, water 56.8%, followed by water 20%, then water 30% and whole eggs 25%. Weigh 350 g batter into two 8 in. pans, bake at 177 C for 30 min. Measure volume (Rape Seed Volumometer), rise (Pillsbury Goal Post) and after halving, observe grain and holes. Results are average of two cakes (four layers).

Toxicity and Irritation Index. Acute oral toxicity in mice was determined according to the method of Weil (7) and of Litchfield and Wilcoxon (8) and judged according to the terminology of Hodge and Sterner (9). Irritation to rabbit skin and eyes was done by the procedure outlined in "Appraisal of the Safety of Chemicals in Foods, Drugs and Cosmetics" (10). Interpretation of scoring was made by the terminology of Kay and Calandra (11) and "The Code of Federal Regulations" (12).

Results and Discussion

Properties

Depending on the alcohol used (mp, decyl alcohol 6 C, *n*-octadecyl alcohol 58 C) and the number of

TABLE XVI

Baked Goods

Glucoside		Bread			Bakers yellow cake, 6% on wt. shortening				
		Volume CC	Softness 6 days	Grain 6 days	Volume CC	Rise	Batter	Grain	Holes
Control		2692	233	Good					
Atmul 500[a]		2717	187	V Good					
n-Decyl,	4.0	2671	217	V Good	2270	+2	Sl gr[b]	Sl irreg.	
n-Hexadecyl,	5.0	2530	248	Good	2105	−2	Sl gr[b]	irreg.	
n-Octadecyl,	8.0	2627	239	V Good	2295	+1	Sl gr[b]	Good	Few small
Atmul 80					2275	0	S & C[c]	Sl irreg.	Few medium
Control		2700	212	Good					
Tween 60[d]		2867	147	V Good					
n-Decyl,	4.0[d]	2800	164	V Good					
n-Hexadecyl,	5.0[d]	2755	175	Good					
n-Octadecyl,	8.0[d]	2785	170	V Good					
Atmul 80		2805	165	Good					
Atmul 80					2325	0	S & C[c]	Sl irreg.	Few medium
Tween 60[e]					2310	−2	S & C[c]	Good	Medium, Small
n-Decyl,	4.0[f]				2245	−2	S & C[c]	Good	Medium, Small
n-Hexadecyl,	5.0[f]				2220	−2	S & C[c]	Good	Few small
n-Octadecyl,	8.0[f]				2290	−1	S & C[c]	Fair	Few medium
Richmix[g]					2210	−2	S & C[c]	Good	Medium, Small

[a] Atlas Chemical Industries, Inc. manufacture Atmul and Tween products.
[b] Slightly grainy.
[c] Smooth and creamy.
[d] Ratio by weight 60:40, Atmul 500/glucoside. (Atmul 500 is mono- and diglycerides from edible fat).
[e] Ratio by weight 80:20, Atmul 80/Tween 60. (Atmul 80 is mono- and diglycerides, Tween 60 is 20 POE sorbitan monostearate).
[f] Ratio by weight 70:30, Atmul 80/glucoside.
[g] HUMKO Products, bakers cake shortening.

glucose units in the molecule (mp D-glucose 146 C), the glucosides vary in appearance from gray, sticky solids to brown, hard waxes and from amber, glassy solids to light-cream powders. Melting range varies from 30 to >300 C and with high glucose containing products, as with many carbohydrates, charring and decomposition takes place without melting. Table I records select properties of a few higher alkyl polyglucosides.

Although the nomenclature of the products (calculated from found hydroxyl number) suggest fairly pure compounds, they are complicated mixtures especially in relation to the glucose units present. Table II shows the results of solvent fractionation of *n*-decyl-4.0-glucoside into 13 fractions. Using the hydroxyl number to calculate glucose units, it will be noted that the calculated units attached to the *n*-decyl radical reach a 4.0 glucose unit peak in Fractions 4, 5 and 6. Molecular weight determinations show (Fractions 2, 4, 10 and 13) an increase in glucose units at the higher fraction level with an overall glucose content at 10.5 units (crude). Careful analyses of starting and recovered materials give a 9.0 glucose units to the glucoside. Although the hydroxyl number analysis does not give an exact glucose unit number, it is an easy and reproducible method of identification.

Comparative foam heights were run on each fraction (Table II) and it will be noticed that foam decreases as the number of glucose units increase. Foam test by the ASTM procedure (Table III) demonstrate that the glucosides produce good stable foams.

The glucosides are in general soluble in water and glycol, but insoluble in most common organic solvents (Table IV). Their aqueous solutions do not exhibit inverse solubility with temperature or concentration; a 50% solution of *n*-decyl-4-glucoside will remain clear at 99 C and at 0 C.

Surface tensions of aqueous solutions are equivalent to surface tensions of commercial surface-active agents (Table V). HLB (Table V) and CMC (Table II) are recorded for a few products.

The viscosity in centipoise of aqueous solutions is given in Table VI. The Brookfield viscometer gives ranges that are higher than those obtained by the ASTM procedure.

Humectant properties are recorded in Table VII. The anhydrous glucosides pick up water slower than a standard humectant and generally lose water faster.

Although the products are nonionic in nature, they are at least as biodegradable as commercial linear alkylaryl sulfonates (Table VIII), and probably more so in view of their chemical composition.

Toxicity studies (Table IX) show that the glucosides are "relatively harmless" (9) when ingested, nonirritating to the skin and from moderate to nonirritating to the eyes.

Detergency

In heavy-duty solid laundry formulations (Table X), the glucosides are equivalent in detergency to commercial products (Ultrawet K) in soft water, but in hard water their efficiency falls to about 85%. Adjustment of the formula might improve their performance in hard water. In heavy-duty liquid formula, the glucosides performed excellently even in hard water. The glucosides are compatible with tetrapotassium pyrophosphate (Table XI), but on addition of sodium carboxymethylcellulose (CMC), the clear solution separates transparent gel specks of CMC. Adjustment of the formula would probably eliminate or minimize the CMC gel specks. Because of the good foam characteristics, water solubility, biodegradability and low toxicity, the glucosides should be good detergents and especially valuable in shampoos.

Perclene soluble alkyl glucosides showed good water solubilization and fair dry cleaning properties; addition of a commercial dry cleaning detergent (G-711) improved the antistatic and soil removal properties but adversely affected the redeposition (Table XII).

Textile Lubricant

The glucosides are poor yarn to yarn lubricants when compared with the commercial product G-263 (Table XIII), but select products do possess good yarn to metal lubricity. No softening effect on cotton fabric was obtained.

Tablet Formation

The products demonstrated good tablet granulating properties (Table XIV) but were of no value as a dry lubricant as they caused the die to bind.

Food Emulsifier

Because of the favorable preliminary toxicity indications the glucosides were examined as surfactants in foods. In ice cream (Table XV) the glucosides produced good body and ribbon but the overrun was not equal to that produced by the commercial product Tween Mos 100VS. The higher glucosides (hexadecyl, octadecyl) produced too much foam so that whipped topping containing them had no body and thus did not peak (Table XV) but a 35% tomato puree did give a fair peak in foam mat drying.

Alone the glucosides do not form a good bread but combined with a mixed glyceride, the baked bread was almost equivalent to bread baked with the standard commercial glyceride Atmul 500 (Table XVI). In cakes the glucosides showed up well when used alone or admixed with glycerides. They were slightly low in volume when compared with the Atmul 80 standard but were at least equivalent to a commercial cake shortening Richmix.

Taste panel tests on the glucosides showed them to be bland and taste acceptable when compared with other food acceptable commercial surfactants.

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