# Preparation and surface active properties of alkyl glucosides of fat Egyptian mangifera indica

# Az Egyiptomban termesztett indiai mangó olaja alkil-glükozidjainak előállítása és felületaktív sajátságaik

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ÖSSZEFOGLALÁS

Mangó-olajból előállított palmitinsavat, sztearinsavat, olajsavat, linolsavat és kevert zsírsavakat metil-észtereikké alakították át, $LiAlH_4$-del a megfelelő zsíralkoholokká redukálva (Ia-e). Az említett zsíralkoholokból kielégítő hozammal alkil-glükozidokat állítottak elő (IIa-e). Az így elkészített alkil-glükozidhoz homogén lúgos közegben propilén-oxidot adagolva nemionos oxipropilezett alkil-glükozidok (IIIa-e) keletkeztek. Ezt a vegyületet klór-szulfonsavval reagáltatták, amelynek során anionos felületaktív sajátságú oxipropilezett alkil-glükozid-szulfát (IVa-e) keletkezett, fokozottabb felületaktivitással, mint az alkil-glükozidok és a propén-oxilezett származékok. Az előállított vegyületek szerkezetét IR és $^1$H NMR spektrumokkal igazolták. Értékelték az előállított detergensek felületaktív sajátságait.

Summary

Hexadecanoic, octadecanoic, octadec-9-enoic, octadec-9,12-dienoic and mixed fatty acids obtained from mangifera oil were converted to their methyl ester, reducing with $LiAlH_4$ to the corresponding fatty alcohols (Ia-e). Alkyl glucosides (IIa-e) from the mentioned fatty alcohols were produced in suitable yield. The adding propylene oxide (PO) to the prepared alkyl glucosides was completed in homogeneous alkaline medium to give nonionic oxypropylated alkyl glucosides (IIIa-e), moreover, the oxypropylated alkyl glucoside was conducted to react with chlorosulphonic acid afforded oxypropylated alkyl glucoside sulphates (IVa-e) as anionic surfactants having prior surface properties to alkyl glucosides and propenoxylated derivatives. The structures of the prepared compounds were confirmed by IR and $^1$H NMR spectra. The surface active properties of the prepared surfactants were evaluated.

ZUSAMMENFASSUNG

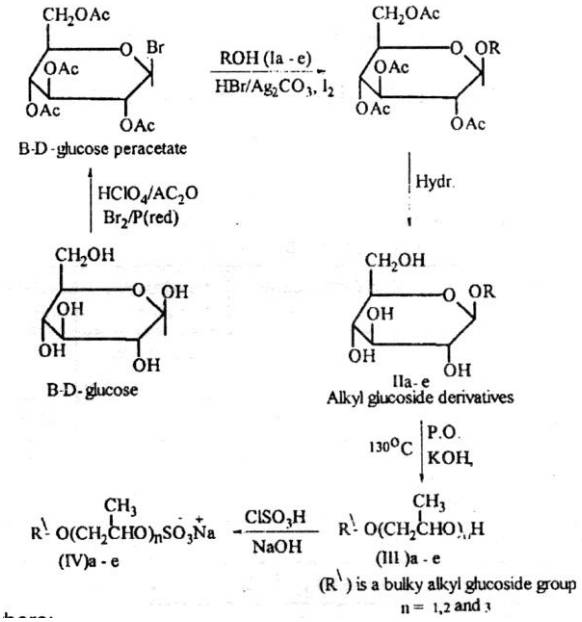
Die aus dem Mangoöl hergestellte Palmitin-, Stearin-, Öl-, Linolsäure, sowie die gemischten Fettsäuren wurden in Methylester umgestaltet, die sich mit der Hilfe von Li Al $H_4$ zu den entsprechenden Fettalkoholen reduziert haben (Ia-e). Aus den erwähnten Fettalkoholen wurden _ mit genügender Ausbeute _ Alkylglykoside hergestellt (IIa-e). Zu den so hergestellten Alkylglykoside wurde in homogenem alkalischem Mittel-Propylenoxyd dosiert und dadurch sind

nicht ionische oxypropylisierte Alkylglykoside (IIIa-e) entstanden. Diese Verbindung wurde mit Chlorsulfonsäure reagiert, im Laufe dessen oxypropylisierte Alkylglykosidsulfonate mit anionischer oberflächenaktiver Eigenschaft (IVa-e) mit gesteigerterer Oberflächenaktivität entstanden sind, als die Alkylglykoside und die propenoxylisierten Folgeprodukten.

Die Struktur der hergestellten Verbindungen wurde IR- und $^{1}$HNMR - Spektren bewiesen. Es wurden die oberflächenaktiven Eigenschaften der hergestellten Detergenten gewertet.

## Introduction

Alkyl glucosides are class of compounds which have been known for some considerable time. In 1893 EmilFischer identified the substance ethyl glucoside as the reaction product of glucose with ethanol in the presence of hydrochloric acid as catalyst [1]. Alkyl glucoside with long alkyl chain (octyl to hexadecyl) was synthesized and their surface properties were evaluated [2-3]. Depending on the particle size of starting glucose, the reaction conditions and modification of the Konigs-Knorr reaction very pure products of predominantly either a- or b-anomer could be synthesized [4, 6]. Because of the lack of a large scale synthesis, the application of alkyl glucoside as surfactants remained restricted for a long time to specified areas, while especially octyl glucoside and dodecyl maltoside have been successfully used in the crystallization of membrane proteins. The surfactants obtained from raw materials acquire increasing interest and shortly produced on an industrial scale. Based on the fact that, mango seed is useless and causing a big problem inside producing factories, authors have planned to utilize it in obtaining intermediate compounds as alkyl glucoside which are widely used in the surface active application field. Moreover, in order to improve their surface properties propylene oxide was added followed by sulphation which afford good surface active properties prior to all synthesized products (cf. Scheme 1.).




where:
R = hexadecyl, octadecyl, octadec-9-enyl, and octadec-9,12-dienyl respectivily.

IIa-e: (hexadecyl, octadecyl, octadec-9-enyl, and octadec-9,12-dienyl, mixed fatty alkyl of fatty alcohol obtained from mangifira oil)

(III)a-e: Alkyl glucoside propenoxilated derivatives

(IV)a-e: Alkyl glucoside propenoxylated sulphated derivatives respectivily

*Scheme 1.*

## Material and Methods

All melting points are uncorrected. The IR Spectrum was measured by Pye-Unicam SR-1000 infra red Spectrophotometer as KBr disk or nujul mul and $^{1}$HNMR was done in DMSO as solvent and tetramethylsilane (TMS) as internal standard [Varian EM-390] Spectrophotometer operating at 90 MHz.

**1. Industrial wastes of mangifera indica**, were kindly supplied by El-Nasr company of canned products, Kaha, Egypt. The oil was extracted from the kernels. The specifications are given in Table I.

**2. Hydrolysis of crude mangifera oil:** The procedure described by El-Sawy et. al. [7]. The fatty acids mixture was analyzed by G. L. C. and its composition is given in Table I.

*Table I*

**Chemical characteristics and fatty acid composition of crude mangifera oil**

| Chemical characterization | | Fatty acid composition as determined by G. L. C. | |
|---|---|---|---|
| | | Fatty acid | Peak % |
| Acid value (A. V) | 26.50 | Saturated fatty acids | |
| | | Palmatic | 12.12 |
| Iodine value (I. V) | 50.22 | Stearic | 36.96 |
| | | Unsaturated fatty acids | |
| Saponification value (S. V.) | 198.07 | Palmitoleic | 0.28 |
| Unsponification (Unsp. V) | 2.06 | Oleic | 47.24 |
| | | Linoleic | 5.40 |

**3. Fatty alcohols [8]:** Were prepared in good yield (79,7-85,7%) by reduction of the corresponding fatty methyl ester (cf. Table II). The products were analyzed for saponification and hydroxyl values according to Rao et. al. [9].

*Table II*

**Characterization of the reduction of methyl ester to corresponding alcohols**

| Compound | Saponification value | | Hydroxyl value | | Reduction % |
|---|---|---|---|---|---|
| | ester | red. product | ester | red. product | |
| Me-hexadecanoate | 199.00 | 36.00 | - | 187.0 | 81.9 |
| Me-octadecanoate | 195.00 | 39.00 | - | 189.0 | 80.0 |
| Me-octadec-9-enoate | 199.50 | 40.22 | - | 198.4 | 79.8 |
| Me-octadec-9,12-dieenoate | 198.00 | 40.00 | - | 192.0 | 79.7 |
| Mixed F.A.Methyl Esters. | 198.00 | 28.16 | - | 197.6 | 85.7 |

*The percent of reduction was calculated according to the following equation: [S.V. of ester - S.V. of reduced product] ´ 100/S.V. of ester*

**4. Fatty Alkyl glucosides:** Were synthesized and purified using the method reported by Rosevear et. al. [6] with several modifications. Typically, 20 gm, of glucose peracetate were dissolved in 50 ml of glacial acetic acid, 50 ml HBr (30% $CH_3COOH$) then added. The light yellow solution produced was stirred at room temp. for 45 min. dichloromethane 200 ml was added and the resulting solution was immediately poured into 300 ml of crushed ice. The dichloromethane layer containing the brominating glucose peracetate was washed once with 200 ml of de-ionized water, three times with 200 ml portions of cooled saturated aqueous sodium

bicarbonate solution and three times with 200 ml of de-ionized water. This layer was finally dried over anhydrous $MgSO_4$ (50 gm) for 30 min. and then filtered. The dichloromethane layer was transferred to flash covered with aluminum fuel and fresh dichloromethane was added to adjust, the total volume to approximately 280 ml. Fatty alcohols (hexadecyl, octadecyl, octadec-9-enyl, octadec-9,12-dienyl and mixed fatty alcohols obtained from the corresponding mixed fatty acids of crude mangifera oil), glucose peracetate by a molar ratio of 1:l, 6 gm of freshly prepared silver carbonate, 0,4 gm iodine, 20 gm of molecular sieves were added. After stirring over night at room temperature, the mixture was filtered slow slight pad and concentrated by using rotary evaporator. Deacetylation was carried out by treatment of the sample with 400 ml of methanol:triethylamine:water:hexane (7:3:1:2) at room temperature for 72 hours [10].

**4.Oxypropylation:** The propylene oxide was added dropwise to alkyl glucoside at 130-140 °C and atmospheric pressure with 4 mole % of potassium hydroxide as catalyst [11]. The steam of nitrogen was pubbled into reaction mixture to remove unreacted propylene oxide. The number of propylene oxide added (n) was counted by increasing in the weight periodically.

**5. Sulphate of oxypropylated alkyl glucoside:** The sulphation of oxypropylated alkyl glucosides (IIIa_e), was carried out according to Chlebick [12].

***6. Surface Properties:***

**6.1. Surface tension and interfacial tension**, were measured using a Du-Nouy tensiometer [13]. (Kruss, Type 8451), with 0,1 wt % aqueous solution at room temperature (25 °C).

**6.2. Cloud Point**, was determined by gradually heating 1,0 wt % solution in controlled temperature bath and recording the time at which the clear, or nearly clear solutions become definitely turbid. The reproducibility of this temperature was checked by cooling the solutions until they become clear again [14].

**6.3. Wetting time:** Was determined by immersing a sample of cotton fabric in 0,1 wt % aqueous solution of the surfactants [15].

**6.4. Foaming properties:** Were measured by Ross Miller method [16]. The foam production for 1,0 wt % solution was measured by the foam height initially produced.

**6.5. Emulsion properties:** The emulsion was prepared from 10 ml of 20 m.mole aqueous solution of surfactant and 5 ml of toluene at 40 °C. The emulsifying property was determined by the time it took for an aqueous volume separating from the emulsion layer to reach 9 ml, counting from the moment of the cession shaking [17].

**6.6. Biodegradability:** The percentage was determined according to Eter et. al. [18].

*Table III*

**Characterization of alkyl glucosides products $II_{a-e}$.**

| Compds No. | Mol. Formula | M wt. | Yield % | Colour | State | m.p °C |
|---|---|---|---|---|---|---|
| IIa* | $C_{22}H_{44}O_6$ | 404 | 39.8 | white | solid | 90-92 |

| | | | | | | |
|---|---|---|---|---|---|---|
| IIb* | $C_{24}H_{48}O_6$ | 432 | 37.5 | white | solid | 93-95 |
| IIc | $C_{24}H_{46}O_6$ | 430 | 40.0 | brow risen | viscous oily | - |
| IId | $C_{24}H_{44}O_6$ | 428 | 36.5 | brow risen | viscous oily | - |
| IIe | - | - | 38.5 | brow risen | viscous oily | - |

* *Crystallized from petroleum ether 40-60 °C*

## Results and discussions

Hexadecyl, octadecyl, octadec-9-enyl, octadec-9,12- dienyl and mixed alkyl of mixed fatty alcohols obtained from the corresponding mixed fatty acid of mangifera oil were prepared in suitable yield (79,7-85,7%) (cf. Table II) [9]. Alkyl glucoside of the target work was synthesized by the condensation of b-bromo D-glucose peracetate and the mentioned fatty alcohols. The products were hydrolyzed to give the corresponding alkyl glucosides (IIa-e) with yields (36,5-40,0%) (cf. Table III) as nonionic surface active agents. The obtained alkyl glucosides were conducted to react with P.O in the presence of potassium hydroxide as catalyst to give different moles (n=1-3) of oxypropylated alkyl glucosides (IIIa-e). The later was sulphated using chlorosulphonic acid to afford good yields of oxypropylated alkyl glucoside sulphates (IVa_e) as anionic surface active agents. The structure of same prepared examples of products was confirmed by IR and $^1$HNMR spectra (cf. Table IV).

*Table IV*

**Spectral data of some examples from the prepared alkyl glucoside and their propenoxylated products**

| Compds | $^1$HNMR (δ = ppm) | IR ($cm^{-1}$) |
|---|---|---|
| (IIa) | δ 0.7 (t, 3H, term.$CH_3$);δ 0.8-1.2 (br.s.; $CH_2$ chain); δ 2.5 (d, 2H, $CH_2$-O sugar); δ 3.1 (br.s.; C-CH-OH) and δ 3.8-4.2 (br.s; 4H, C-OH). | The spectra of all samples show the following data: 3420-3280 $cm^{-1}$ $_{OH}$; 1250, 1150 $cm^{-1}$ $\nu_{c\text{-}o\text{-}c}$; 2970, 2920 $cm^{-1}$ $\nu_{aliphatic\ protons}$ |
| (IIc) | δ 0.8 (t, 3H, term.$CH_3$); δ 0.9-1.1 (br.s.; $CH_2$ chain); δ 1.6 (d, 2H, $CH_2$-HO sugar); δ 3.4 (t, 2H, O-$CH_2CH_2$-) δ 3.7-4.1 (br.s; 4H, C-OH); and δ 5.4 (m, 2H, CH=CH- olefine). | |
| (IId) | δ 0.9 (t, 3H, term.$CH_3$); δ 1.0-1.4 (br.s.; $CH_2$ chain); δ 2.0 (br.s, 2H, $CH_2$-OH sugar); δ 3.4 (t; 2H, O-$CH_2$-$CH_2$-) δ 3.6-4.1 (br. s; 4H, C-OH); and δ 5.4 (br. s, 4H, OH); and δ 5.4 (br. s., 4H olefinic protons). | |
| (IIe) | δ 0.7 (t, 3H, term. $CH_3$) δ 0.8-1.2 (br.s, $CH_2$ chain); δ 2.2 (br.s; 2H, $CH_2$-O) δ 3.1-3.3 (br, s, 4H, CHOH in glucose); δ 3.5 (t, 2H, O-$CH_2CH_2$-); δ 3.6-4.3 (br. s, 4H, CH-OH); and δ 4.9 (br. s, olefinic protons). | 3500-3300 $cm^{-1}\nu_{OH}$, 3090 $cm^{-1}$ $\nu_{CH\ olifinic}$, 2980, 2920 $cm^{-1}$ $\nu_{CH\ aliphatic}$, and 1640 $cm^{-1}\nu_{C=C}$, 1450, 1350, 1150 $cm^{-1}$ $\nu_{C\text{-}O\text{-}C}$, |
| The prepared propenoxylated alkyl glucosides has identical charactesristic bands in $^1$HNMR, the example is: | | |
| IIIe | δ 0.8 (t, 3H, term. $CH_3$) δ 0.9 (d, $CH_3$ of P.O.); δ 1-1.3 (br.s; $CH_2$ chain) δ 2.3 (br, s, 2H, $CH_2$O); δ 3.0 (t, 2H, O-$CH_2CH_2$); δ 3.2-3.7 (m, O-$CH_2$CH in P.O.); δ and δ 4.1-4.3 (br. s, OH). | |

### *1. Surface active properties*

The measuring data of the prepared alkyl glucosides and glucoside derivatives were tabulated in the Tables V_VII.

**1.1. Surface and interfacial tensions:** Surface active properties of the prepared b-D-glucoside of

hexadecyl, octadecyl, octadec-9-enyl, octadec-9,12-dienyl and mixed fatty alkyl glucoside, its propenoxylated and sulphated derivatives were evaluated. It was found that, in all prepared cases, the compound carries hexadecyl hydrophobic moiety has a good surface activity and records the lower values of surface and interfacial tensions than that all other prepared compounds. On other hand, the prepared series of mixed fatty alkyl glucosides of mangifera oil show the higher values, oxypropylation of alkyl glucoside causes an increasing in surface tension as shown in Table VI. The sulphation of propenoxylated alkyl glucosides improves their surface and interfacial tensions as in Table VII, the presence of the sulphate group in the compounds allowed its to dissociate electrically and dissolve well in water [19]. Finally, surface and interfacial tensions depends on the length and structure of the molecule or also on the kind of hydrophilic group.

*Table V*

**Surface properties of alkyl glucoside surfactants $II_{a-e}$**

| compds | S.T. (dyne/cm) 0.1wt% | I.F.T (dyne/cm) 0.1wt % | wetting time 0.1wt % (sec.) | foam height 0.1wt % (mm) | cloud point 0.1wt % (°C) | emul. stab. 20 m. mole min : sec |
|---|---|---|---|---|---|---|
| Ila | 42.5 | 7.5 | 160 | 140 | 72 | 238 : 18 |
| Iib | 46.0 | 11.0 | 185 | 150 | 69 | 242 : 00 |
| Iic | 44.5 | 9.5 | 190 | 155 | 81 | 245 : 00 |
| Iid | 54.0 | 12.5 | 198 | 120 | 84 | 248 : 00 |
| Iie | 47.0 | 11.5 | 202 | 110 | 90 | 253 : 00 |

*Table VI*

**Surface properties of mixed and prepared oxypropenoxylated alkyl glucoside $III_{a-e}$**

| compds | mole of P.O. | S.T. (dyne/cm) 0.1wt% | I.F.T (dyne/cm) 0.1wt % | wetting time 0.1wt % (sec.) | foam height 0.1wt % (mm) | cloud point 0.1wt % (°C) | emul. stab. 20 m. mole min : sec |
|---|---|---|---|---|---|---|---|
| IIIa | 1 | 44.5 | 09.0 | 160 | 190 | 71.0 | 215 : 12 |
| | 2 | 45.0 | 10.5 | 155 | 190 | 77.0 | 207 : 22 |
| | 3 | 46.0 | 12.0 | 140 | 175 | 84.0 | 201 : 15 |
| IIIb | 1 | 46.0 | 10.0 | 173 | 170 | 70.0 | 232 : 02 |
| | 2 | 47.0 | 11.5 | 159 | 160 | 72.0 | 227 : 55 |
| | 3 | 47.0 | 13.0 | 155 | 150 | 77.0 | 219 : 43 |
| IIIc | 1 | 47.0 | 11.5 | 179 | 200 | 80.0 | 247 : 12 |
| | 2 | 47.5 | 12.0 | 173 | 195 | 87.0 | 239 : 12 |
| | 3 | 48.0 | 13.5 | 167 | 180 | 88.0 | 235 : 25 |
| IIId | 1 | 48.0 | 12.0 | 178 | 215 | 85.5 | 243 : 15 |
| | 2 | 48.5 | 13.0 | 168 | 205 | 87.0 | 239 : 16 |
| | 3 | 49.5 | 14.0 | 171 | 195 | 87.0 | 238 : 36 |
| IIIe | 1 | 48.5 | 12.5 | 198 | 220 | 90.0 | 245 : 15 |
| | 2 | 49.0 | 13.5 | 185 | 195 | 92.0 | 239 : 43 |
| | 3 | 49.5 | 14.5 | 183 | 180 | 95.5 | 231 : 26 |

**1.2. Cloud and Kraft points:** Generally, the cloud point increases by increasing the hydrophobic part in the molecule. The prepared nonionic alkyl glucoside compounds shows cloud points higher than 69°C Table V. Somewhat effect was happened by insertion of propylene oxide units

in the alkyl glucoside molecules (cf. Table VI).

Anionic alkyl glucoside sulphate exhibited Kraft point, also propylene oxide units has some effect on it (cf. Table VII).

**1.3. Wetting properties:** the measuring data of the wetting time from the prepared alkyl glucoside was recorded in (cf. Table V), hexadecyl glucoside recorded lower wetting time value rather than the other prepared alkyl gluco

*Table VII*

**Surface properties of mixed and prepared alkyl glucoside propenoxylated sulphate $IV_{a-e}$**

| compds | mole of P.O. | S.T. (dyne/cm) 0.1wt% | I.F.T (dyne/cm) 0.1wt % | wetting time 0.1wt % (sec.) | foam height 0.1wt % (mm) | emul. stab. 20 m. mole sec |
|---|---|---|---|---|---|---|
| IVa | 1 | 35.5 | 07.5 | 75.0 | 260 | 625 |
| | 2 | 36.0 | 09.0 | 63.0 | 250 | 612 |
| | 3 | 37.5 | 10.5 | 52.0 | 240 | 599 |
| IVb | 1 | 36.5 | 08.0 | 78.0 | 250 | 665 |
| | 2 | 38.0 | 10.0 | 67.0 | 250 | 653 |
| | 3 | 38.5 | 12.0 | 59.0 | 240 | 643 |
| IVc | 1 | 37.0 | 08.5 | 77.0 | 270 | 710 |
| | 2 | 39.0 | 10.0 | 69.0 | 265 | 699 |
| | 3 | 39.5 | 12.5 | 63.0 | 255 | 619 |
| IVd | 1 | 37.5 | 09.5 | 80.0 | 265 | 713 |
| | 2 | 38.0 | 12.5 | 78.0 | 260 | 672 |
| | 3 | 40.5 | 13.0 | 73.0 | 260 | 653 |
| IVe | 1 | 39.0 | 11.5 | 105.0 | 270 | 721 |
| | 2 | 40.5 | 12.0 | 97.0 | 260 | 693 |
| | 3 | 41.5 | 12.5 | 82.0 | 245 | 685 |

*Table VIII*

**Biodegradability of the mixed and prepared alkyl glucoside $II_{a-e}$**

| compds | $1^{st}$ | $2^{nd}$ | $3^{th}$ | $4^{th}$ | $5^{th}$ |
|---|---|---|---|---|---|
| Ila | 58.5 | 69.5 | 81 | 94 | - |
| Ilb | 57.5 | 66.0 | 79 | 87 | 97 |
| Ilc | 61.0 | 78.5 | 92 | 97.5 | - |
| Ild | 64.0 | 77.5 | 89.5 | 96.5 | - |
| Ile | 59.5 | 67.5 | 85.5 | 94.0 | - |

side compounds. However the insertion of different units (1-3) of propylene oxide in the alkyl glucoside molecules causes reduction of wetting time i.e. improves their wetting properties [19] as in Table VI. More reduction of the wetting time achieved by sulphation of these compounds (cf. Table VII).

**1.4. Foaming height:** The foaming power of the prepared compounds was investigated using the Ross-Miler method. All the prepared alkyl glucosides show low foam as nonionic surface active

agents (cf. Table V). The value of foam height in general decreased by increasing propylene oxide unit in the alkyl glucoside molecule, the alkyl radical length remaining constant [19] Table VI. However, the foaming height improved by sulphation, but the same trend was observed (cf. Table VII).

**1.5. Emulsification stability:** The emulsification stability was determined using standard procedure [17]. The data in Tables V_VI reveal that the alkyl glucosides posses greater emulsifying stability than propenoxylated [11] and sulphated compounds. Therefore the prepared alkyl glucoside compounds acts as a good emulsifying agents (cf. Table V).

**1.6. Biodegradability properties:** The course of degradation in river die-away tests was followed by foam and surface tension measurements. The pertinent data for prepared alkyl glucoside and its propenoxylated are given in Table VIII and IX respectively. The results reflect that, within the experimental accuracy, all prepared and mixed alkyl glucosides seem to degrade more easier than that its propenoxylated products. In side the same type of compound products, the variation in the rate of degradation results from the difference in the number of the carbon atom in the molecule. This by means that biodegradability decreased with increasing the number of the carbon atom, led to the conclusion that, a longer alkyl chain makes the diffusion of the molecule through the cell membrane, moreover the degradation more difficult. The same results were achieved with mixed alkyl glucoside compounds.

*Table IX*

**Biodegradability of the mixed and prepared oxypropenoxylated alkyl glucoside $III_{a\_e}$**

| compds | P.O. | 1st | 2nd | 3th | 4th | 5th | 6th | 7th |
|---|---|---|---|---|---|---|---|---|
| IIa | 1 | 52.0 | 60.5 | 70.5 | 77.0 | 82.5 | 88.0 | 92.5 |
| | 2 | 49.5 | 59.5 | 69.0 | 74.5 | 79.5 | 87.5 | 93.0 |
| | 3 | 48.0 | 58.0 | 65.5 | 74.5 | 79.0 | 87.5 | 92.0 |
| IIb | 1 | 53.0 | 60.0 | 69.5 | 76.5 | 81.5 | 90.5 | 97.0 |
| | 2 | 49.0 | 59.5 | 68.0 | 75.0 | 79.0 | 89.5 | 95.0 |
| | 3 | 49.0 | 59.0 | 67.0 | 75.0 | 78.5 | 88.0 | 91.0 |
| IIc | 1 | 59.5 | 69.0 | 77.5 | 83.0 | 89.5 | 92.5 | 97.5 |
| | 2 | 58.0 | 67.0 | 73.5 | 79.5 | 86.5 | 91.0 | 95.0 |
| | 3 | 57.0 | 63.5 | 73.0 | 78.0 | 85.0 | 91.0 | 93.0 |
| IId | 1 | 58.5 | 67.5 | 75.5 | 78.0 | 88.0 | 94.0 | - |
| | 2 | 56.0 | 66.5 | 73.0 | 79.0 | 86.5 | 92.0 | 94.0 |
| | 3 | 55.5 | 64.5 | 71.0 | 78.0 | 85.0 | 92.0 | 92.0 |
| IIe | 1 | 59.0 | 65.0 | 77.0 | 82.0 | 88.5 | 92.0 | - |
| | 2 | 56.0 | 64.5 | 74.0 | 78.0 | 85.0 | 91.5 | 97.0 |
| | 3 | 53.0 | 62.2 | 71.5 | 77.0 | 81.5 | 88.5 | 94.5 |

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