

# Preparation and Surface Active Properties of Novel Succinic Acid Based Surfactants

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## ÖSSZEFOGLALÁS

A dikarboxilsav-anhidrideket felületaktív anyagok gyártására használják. Ezek között a borkősav-anhidridet is jó kiinduló anyagnak tartják. Ebből az anyagból három nem ionos típust állítottak elő. 1. Etoxilezett zsíralkoholokkal ( $C_8-C_{18}$  3–10 mól etilén-oxiddal) diészter típusú nem ionos felületaktív anyagot ad. 2. Itt a nem ionos felületaktív anyag hidrofíli részét zsíralkohol ( $C_6-C_{18}$ )-szukcinát félészterrel etoxilálva készítették el  $Na_2CO_3$  katalizátor jelenlétében. 3. Amidálással (etanol-aminnal) és a félészter etoxilezésével K10 derítőföld katalizátorral. Az új, nem ionos felületaktív anyagok szerkezetét spektroszkópiás vizsgálatokkal igazolták, és meghatározták felületaktív sajátságait és biológiai lebonthatóságukat. Az összes nem ionos terméknek jó felületaktív tulajdonságai voltak. Az azonos szubsztátumot és azonos számú etilén-oxidot tartalmazó nem ionos termékek közül a K10 derítőföldes katalizátorral gyártottaknak voltak a legjobb felületaktív sajátságai.

**Kulcsszavak:** felületaktív anyagok, borkősav-anhidrid, nem ionos felületaktív anyagok, biológiai lebonthatóság

## ABSTRACT

The dicarboxylic anhydrides are used in preparation of surfactants. Among these succinic anhydride is considered as a good precursor, or starting material for production of surface active agents. Three types of nonionic surfactants were prepared with this compound. 1) Succinic anhydride was esterified with ethoxylated fatty alcohols ( $C_8-C_{18}$  with 3–10 mole of ethylene oxide) producing diester type nonionic surfactant. 2) The hydrophilic part of nonionic surfactants was produced by ethoxylation of fatty alcohol ( $C_6-C_{18}$ ) succinate half ester in the presence of  $Na_2CO_3$  catalyst. 3) or by amidation (with ethanol amine) and ethoxylation of the half ester in the presence of K10 clay catalyst. The structures of the prepared new nonionic surfactants were confirmed by spectroscopic tools, their surface properties and biodegradability were determined. All types of nonionic surfactants prepared have good surface properties. The nonionics produced with K10 clay catalyst has superior surface properties compared to others having the same number of ethylene oxide and substrate.

**Keywords:** surfactants, tartaric acid anhydride, nonionic surface active agents, biodegradability

## ZUSAMMENFASSUNG

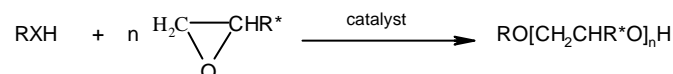
Dikarbonanhydride werden für Herstellung von grenzflächenaktiven Substanzen verwendet. Zwischen diesen Verbindungen ist Succinatanhydrid für ein gutes Ausgangsmaterial für die Herstellung von grenzflächenaktiven Substanzen betrachtet. Aus dieser Verbindung wurden drei Gruppen der nichtionischen grenzflächenaktiven Verbindungen hergestellt. 1. Succinatanhydrid wurde mit äthoxylierten Fettalkoholen ( $C_8-C_{18}$  mit 3–10 Mol Äthylenoxid) verestert und so grenzflächenaktive Verbindungen vom Diester Typ hergestellt. 2. Der hydrophile Teil der nichtionischen grenzflächenaktiven Verbindung wurde durch Äthoxylierung mit Fettalkohol ( $C_6-C_{18}$ )succinat und mit  $Na_2CO_3$  Katalysator hergestellt. 3. Mit Amidierung (Äthanolamine) und Äthoxylierung des Halbesters in der Anwesenheit von K10 Bleicherdekatalysator. Die Struktur der neuen nichtionischen grenzflächenaktiven Verbindungen wurde mit spektroskopischen Untersuchungen bestätigt, und wurden ihre grenzflächenaktive Eigenschaften und biologische Abbaubarkeit auch festgelegt. Alle nichtionische Produkte hatten gute grenzflächenaktive

Eigenschaften. Von den nichtionischen Produkten, die das gleiche Substrat und gleichzählige Äthylenoxid enthielten hatten die mit K10 Katalysator hergestellten Produkte die besten grenzflächenaktiven Eigenschaften.

**Schlüsselwörter:** grenzflächenaktive Substanzen, Weinsäureanhydrid, nichtionische grenzflächenaktive Substanzen, biologische Abbaubarkeit

## 1. Introduction

One of the most important groups of surfactants with growing industrial interest is the nonionics, which can be synthesized by hydroxyalkylation (ethoxylation with ethylene or propoxylation with propylene oxide) of hydrophobic organic compounds containing active hydrogen in the presence of traditional catalyst (conventional basic catalysts, e.g. NaOH, or acids e.g.  $BF_3$ , e.t.c.[1, 2], rather than untraditional catalyst e.g. different clays, calcium compounds, e.t.c.) [3, 4].



where R is long chain aliphatic hydrocarbon, alkyl phenyl, aralkyl group

XH is OH, SH, COOH, NH, e.t.c.

R\* is H (ethoxylation) or  $CH_3$  (propoxylation)

n is moles of alkylene oxide reacted with one mole of starting material

The product of the hydroxyalkylation is a mixture of oligo(poly)ethylene glycol ethers (or esters). Therefore the properties of nonionics depend on the alkylene oxide consumed and the type of starting molecules.

The hydrophilic group generally is a oligo(poly)ethylene glycol chain with a free hydroxyl group at the end of molecule. But in the last time, this free hydroxyl group is chemically bonded. By this way new types of surfactants with better (or different) properties are produced. One possibility for the blocking of hydroxyl group is the usage of dicarboxylic acids in an esterification reaction. By this way (after sulfonation) sulfosuccinate half ester anionic surfactants can be produced, which have good foam and viscosity properties [5]. Two headed surfactants (disodium-4-alkyl-3-sulfosuccinate) were synthesized by the monoesterification of maleic anhydride with fatty alcohols ( $C_6-C_{10}$ ) and addition of sodium bisulfate to the corresponding monoesters. Their micellar properties were studied and interpreted in comparison with a single-headed, single tailed surfactant [6, 7]. Monoester sulfosuccinate does not irritate the eyes and skin in shampoos [8, 9]. The sulfosuccinate surfactants have good surface properties [10, 11 and 12].

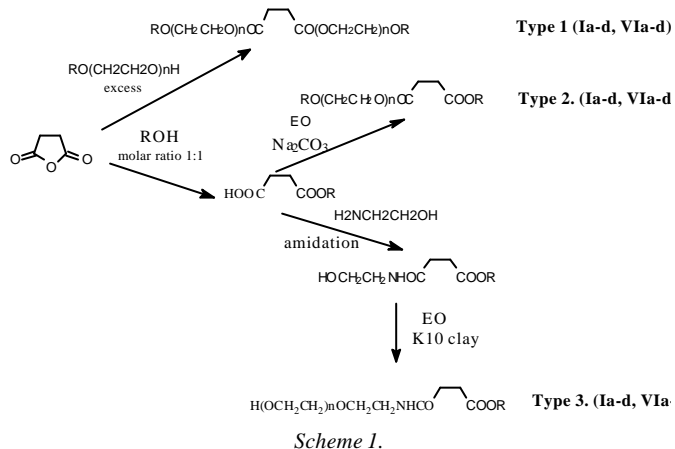
According new research works in the field of preparation of nonionics having no hydroxyl group in the molecule can be prepared by direct hydroxyethylation of fatty acid methyl es-

ters using selected special catalysts [4, 13 and 14]. These long-chain aliphatic esters as nonionic surfactants have good surface properties [15].

The catalyst for esterification of hexyl alcohol with maleic anhydride was p-toluene sulphonic acid [16]. The presence of ester group has a pronounced effect on the behavior of surface properties of a surfactant molecule [17].

The aim of our work was to prepare nonionic surfactants in which the hydrophilic part of the surfactant is in the middle of the molecule using succinic anhydride as base material. This work deals with preparation of different series of nonionic surfactants by using different catalysts and hydrophobic groups in the reaction with succinic anhydride as substrate. The surface properties of these novel surfactants were investigated.

The reactions of the preparation and the achieved types of surfactants are shown in Scheme 1.



R = octyl, decyl, dodecyl, tetradecyl and octadecyl,  
n (number of ethylene oxide moles/starting material moles) = 3, 5, 7 and 10 (a-d)

## 2. Experimental

### 2.1. Hydroxyethylation of fatty alcohols:

It was carried out as described in [18], and the amount of ethylene oxide uptake was determined from the increasing in the mass of the reaction mixture and was controlled by spectroscopic tools [19]. The conditions of hydroxyethylation are in Table 1.

Table 1.

Reaction conditions of hydroxyethylation process.				
Substrate	Catalysts wt%	Temperature °C	Moles of ethylene oxide uptake (n)	Surfactant
Fatty Alcohol	NaOH, 0.1 wt%	120–130	3, 5, 7, 10	Used in surfactants type 1.
Succinic acid half esters	Na <sub>2</sub> CO <sub>3</sub> , 0.1 wt%	80–100	3, 5, 7, 10	Type 2
Succinic half ester amide	K10 clay, 0.1 wt%	40–50	3, 5, 7, 10	Type 3.

### 2.2. Preparation of nonionic surfactants type 1 (I a-d, -VIa-d)

Succinic anhydride was esterified with ethoxylated alcohols prepared from above step at 120–130 °C. A typical procedure: of ethoxylated octyl alcohol (n = 5, 0.02 mol, 3.49 g) and of

Table 2.

Reaction conditions of the preparation of succinic half esters				
Substrate	Alcohols (mol, g)	Products (g)	m.p °C	Yield %
Succinic anhydride 0.02 mol, 2.0 g	Octanol, 0.02, 2.6	Oily 3.68	–	80
	Decyl, 0.02, 3.16	Waxy, 3.5	–	68
	Dodecyl, 0.02, 3.8	Waxy solid, 3.9	–	68
	Tetradecyl, 0.02, 3.8	White solid, 3.9	54–55	65
	Hexadecyl, 0.02, 4.8	White solid, 3.7	60–61	60
	Octadecyl, 0.02, 5.4	White solid, 4.3	77–78	58

succinic anhydride (0.01 mol, 1.0g.) were refluxed in dry benzene, for 10 h, until theoretical amount of water liberated (by using a Dean and Stark apparatus). The organic solvent was removed under reduced pressure and the product was collected with good yield (60–80%) (identification by spectroscopic tools c. f. Table 3).

Table 3.

Spectral data of new prepared compounds	
Compd. No.	<sup>1</sup> HNMR (δ = ppm, CDCl <sub>3</sub> , 90 MHz) IR (cm <sup>-1</sup> )
<b>Type 1.IIIb:</b>	
<b>2(C12, 5 EO):</b>	d 0.80 (t, 6H, term. 2CH <sub>3</sub> ); 2890–2880 cm <sup>-1</sup> n CH ali.
	d 1.2–1.7(m, 44H, -22CH <sub>2</sub> -chain); 1737–1720 cm <sup>-1</sup> n CO of ester
	d 2.8 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO); 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
	d 4.3 (t, 4H, 2-CH <sub>2</sub> -OCO);
	d 3.5–3.8 (m, 36H, 2[-OCH <sub>2</sub> CH <sub>2</sub> (CH <sub>2</sub> CH <sub>2</sub> O) <sub>n</sub> ])
<b>VI d: 2(C18, 10EO):</b>	d 0.89 (t, 6H, term. 2CH <sub>3</sub> );
	d 1.4–2.1(m, 68H, -34CH <sub>2</sub> -chain); 2990–2880 cm <sup>-1</sup> n CH ali.
	d 2.8 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO);
	d 4.3 (t, 4H, 2-CH <sub>2</sub> -OCO); 1737–1720 cm <sup>-1</sup> n CO of ester
	d 3.6–3.8 (m, 76H, 2[-OCH <sub>2</sub> CH <sub>2</sub> (CH <sub>2</sub> CH <sub>2</sub> O) <sub>n</sub> ]) 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
<b>Type 2.</b>	
<b>Ia: (C8, 3 EO):</b>	d 0.88 (t, 3H, term. CH <sub>3</sub> ); 3500–3400 cm <sup>-1</sup> n OH.
	d 1.1–1.3(m, 12H, -6CH <sub>2</sub> -chain); 2880–2870 cm <sup>-1</sup> n CH ali.
	d 2.8 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO); 1735–1720 cm <sup>-1</sup> n CO of ester
	d 4.1 (t, 4H, 2-CH <sub>2</sub> -OCO); 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
	d 3.4–3.6 (m, 10H, [-OCH <sub>2</sub> CH <sub>2</sub> (CH <sub>2</sub> CH <sub>2</sub> O) <sub>2</sub> ]) 3500–3400 cm <sup>-1</sup> n OH.
<b>Vc: (C16, 7 EO):</b>	d 0.80 (t, 3H, term. CH <sub>3</sub> );
	d 1.2–1.5 (m, 28H, -14CH <sub>2</sub> -chain); 2880–2870 cm <sup>-1</sup> n CH ali.
	d 2.7 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO); 1735–1720 cm <sup>-1</sup> n CO of ester
	d 4.2 (t, 4H, 2-CH <sub>2</sub> -OCO); 1650 cm <sup>-1</sup> n CO of amide
	d 3.4–3.6 (m, 26H, [-OCH <sub>2</sub> CH <sub>2</sub> (CH <sub>2</sub> CH <sub>2</sub> O) <sub>n</sub> ]) 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
<b>Type 3.</b>	
<b>Vc: (C16, 7 EO):</b>	d 0.80 (t, 3H, term. CH <sub>3</sub> );
	d 1.2–1.5 (m, 28H, -14CH <sub>2</sub> -chain); 2880–2870 cm <sup>-1</sup> n CH ali.
	d 2.7 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO); 1735–1720 cm <sup>-1</sup> n CO of ester
	d 4.2 (t, 2H, -CH <sub>2</sub> -OCO); 1650 cm <sup>-1</sup> n CO of amide
	d 3.4–3.6 (m, 30H, [-NCH <sub>2</sub> CH <sub>2</sub> O(CH <sub>2</sub> CH <sub>2</sub> O) <sub>2</sub> ]) 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
	d 4.3–4.1 (br., 1H, OH); and
	d 4.0 (t., 2H, CH <sub>2</sub> NCO). 3500–3350 cm <sup>-1</sup> n OH.
<b>IIa: (C10, 3 EO):</b>	d 0.80 (t, 3H, term. CH <sub>3</sub> );
	d 1.2–1.5 (m, 16H, -8CH <sub>2</sub> -chain); 2880–2870 cm <sup>-1</sup> n CH ali.
	d 2.7 (m, 4H, -OCOCH <sub>2</sub> CH <sub>2</sub> -OCO); 1735 cm <sup>-1</sup> n CO of ester
	d 4.2 (t, 2H, -CH <sub>2</sub> -OCO); 1660 cm <sup>-1</sup> n CO of amide
	d 3.4–3.6 (m, 14H, [-NCH <sub>2</sub> CH <sub>2</sub> O(CH <sub>2</sub> CH <sub>2</sub> O) <sub>2</sub> ]) 1100–1120 cm <sup>-1</sup> n of CH <sub>2</sub> OCH <sub>2</sub>
	d 4.3–4.1 (br., 1H, OH); and
	d 4.0 (t., 2H, CH <sub>2</sub> NCO). 3500–3400 cm <sup>-1</sup> n OH.

### 2.3. Preparation of half esters

A typical procedure: of succinic (0.1 mol, 10.0 g) anhydride and hexadecyl alcohol (0.1 mol, 24.2 g) were placed into 150 cm<sup>3</sup> round-bottomed flask fitted with a reflux condenser. The reaction mixture was stirred and heated in oil bath at 100 °C for 20 h. The yield and the physical properties of product half esters can be seen in Table 2. The structure of the yield product was confirmed by spectroscopic data (c.f. Table 3).

### 2.4 Preparation of nonionic surfactants type 2 (Ia–d–VIa–d)

The preparation of nonionic surfactants was completed by hydroxyethylation of prepared half ester (2.3). The catalyst of the procedure was sodium carbonate, (c.f. Table 1.).

### 2.5. Amidation of half esters

Typical procedure: succinic half ester of dodecyl alcohol (0.022 mol, 6.29 g), ethanolamine (0.033 mol, 2.01 g) and molecular sieve 4Å (5 g.) was heated at 120 °C for 19 h. in dry xylene. The organic layer was filtered and the crude was used without purification to next step.

### 2.6 Preparation of nonionic surfactants type 3 (Ia–d–VIa–d)

The preparation of nonionic surfactants was completed by hydroxyethylation of the hydroxyl group in the half ester amide. The catalyst of the procedure was as mentioned K10 clay catalyst (c.f. Table 1.).

### Analytical procedures

All melting points are corrected. The IR spectra were measured by a Pye-Unicam SR 1000 spectrophotometer in film on KBr disk or in Nujol mull using.

Nuclear magnetic resonance spectra (<sup>1</sup>HNMR) were recorded on a Varian EM-390 spectrometer operating at 90 MHz in deuterio-chloroform and tetramethyl silane as internal standard. Chemical shifts are reported as (ppm) relative to tetramethyl silane.

## 3. Surface Properties of the prepared nonionic surfactants

The surface properties were measured under neutral conditions, in aqueous solution of ethoxylated compounds.

**3.1 The surface and interfacial tensions** were measured by a Du-Nouy Tensiometer (Kruss, type 8451) at surfactant concentration 0.1 wt % and at room temperature 25 °C, using [20]

**3.2 Cloud Point** was determined by gradually heating of the prepared surfactant solution (1.0 wt % concentration) 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 [21].

**3.3 Wetting time** was determined by immersing a sample of cotton fabric (3 g. on a stainless steel hook) in 0.1 wt % aqueous solutions of the surfactants at 25 °C. The recorded time from the moment the cotton was put into the solution until the moment it stated going down is wetting time [22].

**3.4 Emulsion stability determination** was prepared from 10 ml of 20 mmol aqueous solution of surfactants and 5 ml of toluene at 40 °C. The emulsion stability was determined as the time of separation of water (9 ml) from the emulsion layer from the moment of the cession shaking [17].

**3.5 Foaming height** was measured according vigorous shaking of 25 ml surfactant solution (0.1 wt %) in 100 ml glass stopper graduated cylinder at 25 °C for 10 seconds. The solution was allowed to stand for 30 seconds and the foam height was measured [23].

**3.6 Hydrophilic Lypophilic Balance (HLB)** of the prepared surfactants were calculated according to Dieves equation [24].

**3.7 Biodegradability in percentage** was determined according to Eter et al. [25].

## 4. Results and Discussion

In the present work, three types of nonionic surfactants were synthesized from precursor succinic anhydride, which is a good starting material due to its bifunctional character. The structures of the surfactants prepared were built up by three different ways. In the first route (for Type I), conventional nonionic surfactants were prepared by ethoxylation of fatty alcohols [octyl, decyl, dodecyl, tetradecyl, hexadecyl and octadecyl, (C8–C18, I–VI)] with various moles of ethylene oxide contents (n = 3, 5, 7 and 10 corresponding to a, b, c and d). The free hydroxyl group of the ethoxylated alcohols was blocked by esterification with succinic anhydride (molar ratio 1: 2, succinic: ethoxylated fatty alcohols) in dry benzene producing succinate diesters of ethoxylated alcohols (nonionic surfactants type 1. (Ia–d – VIa–d) c. f. Scheme 1) In this type two hydrophilic ethylene glycol chains flanked between two hydrophobic long chain alkyl groups in the succinate moiety. The structures of the prepared surfactants were confirmed by spectroscopic methods (c.f. Table 3.). The most characteristic IR spectra bands in these structures are  $\nu_{C=O}$  of esters appeared at 1735  $cm^{-1}$  and  $\nu_{C-O-C}$  of glycol ethers at 1100–1120  $cm^{-1}$ .

In type 2 (Ia–d – VIa–d) the nonionic surfactants were constructed directly by monoesterification of succinic anhydride with fatty alcohols (C8–C18) (molar ratio 1:1). The most characteristic IR bands are  $\nu_{C=O}$  of bath acid at 1710 and ester at 1735  $cm^{-1}$  and OH of acid at 3300–2600  $cm^{-1}$ . The free carboxyl group in the succinate half fatty alcohol ester was ethoxylated using sodium carbonate as catalyst producing **nonionic surfactants** with n = 3, 5, 7 and 10. The structures of these types were confirmed by **spectroscopic methods** (c. f. Table 3). In this type of the surfactant molecules the hydrophilic group is at terminal position.

In Type III, we wanted to achieve a more stable connection between the succinic part and the hydrophilic glycol chain. Amides with free hydroxyl group were prepared by the reaction of half ester and ethanol amine. The structures of these compounds were confirmed by IR (two carbonyl groups: one for ester at 1735 and another for amide at 1650  $cm^{-1}$  and also NH bands at 3000–3100  $cm^{-1}$ ). The glycolic chain was introduced by ethoxylation of the free hydroxyl in the amide using K10 clay catalyst which makes narrow range distribution [18].

### 4.1 Surface properties of the prepared surfactants

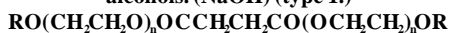
The surface properties of the prepared succinic acid based surfactants has been measured in neutral medium by traditional procedures. The resulted data are given in Tables 4, 5 and 6.

#### 4.1.1 The surface and interfacial tensions

They were determined according to Findly [20]. The measured surface and interfacial tensions data given in Table 4, 5 and 6. The values follow the general term, that the surface and interfacial tensions were increased with increasing number of ethylene oxide units added to the molecule and also with increasing in the number of carbon atoms in the hydrophobic part at same ethylene oxide units [26]. Also it appeared that, the Type I surfactants have the highest surface tensions i.e. low surface activity and the Type III surfactants have the lowest surface tensions (high surface activity).

Table 4:

Surface properties of synthesized succinate diesters with ethoxylated alcohols. (NaOH) (type 1.)



Comp.	n	Surface Tension (dyne/cm) 0.1 wt%	Interfacial tension (dyne/cm) 0.1 wt%	Cloud Point °C 1.0 wt%	Wetting time (sec.) 0.1 wt%	Emulsion stability (min:sec) 20m.mole	Foam height (mm) 0.1 wt%	Calc. HLB
<b>C8 a</b>	3	30.0	10.0	60.0	45.0	65:00	60	11.62
<b>b</b>	5	31.5	10.5	62.0	40.0	66:00	70	13.50
<b>c</b>	7	33.0	11.0	65.0	38.0	70:00	75	14.69
<b>d</b>	10	34.0	12.5	70.0	35.0	75:58	80	15.84
<b>C10 a</b>	3	32.0	11.0	63.0	50.0	64:00	70	10.63
<b>b</b>	5	33.5	11.5	65.0	48.0	65:55	72	12.60
<b>c</b>	7	34.0	12.0	68.0	45.0	68:00	77	13.88
<b>d</b>	10	35.0	13.0	72.0	43.0	72:00	85	15.15
<b>C12 a</b>	3	33.0	12.5	68.0	54.0	60:25	75	9.81
<b>b</b>	5	34.0	13.0	70.0	52.0	62:00	78	11.81
<b>c</b>	7	35.0	13.5	72.0	50.0	64:32	82	13.20
<b>d</b>	10	36.5	14.0	75.0	50.0	68:00	85	14.51
<b>C14 a</b>	3	35.0	12.5	70.0	60.0	50:05	78	9.09
<b>b</b>	5	36.5	14.0	72.0	58.0	52:00	80	11.11
<b>c</b>	7	37.0	14.5	73.0	55.0	55:45	85	12.50
<b>d</b>	10	38.0	15.0	75.5	53.0	58:00	88	13.92
<b>C16 a</b>	3	35.0	13.0	72.0	63.0	40:25	78	8.48
<b>b</b>	5	37.5	14.5	74.0	60.0	43:24	82	10.49
<b>c</b>	7	39.0	15.0	75.0	59.0	45:00	89	11.91
<b>d</b>	10	40.0	16.0	78.0	55.0	50:11	95	13.38
<b>C18 a</b>	3	36.0	13.5	82.0	69.0	30:09	80	7.94
<b>b</b>	5	38.5	14.5	84.0	65.0	30:00	88	9.94
<b>c</b>	7	41.0	15.5	88.0	63.0	35:36	93	11.37
<b>d</b>	10	42.0	16.0	90.0	60.0	38:58	100	12.88

#### 4.1.2 Cloud points

The data (given in Table 4, 5 and 6) show that the cloud point increases with increasing hydrophobic part and with increasing ethylene oxide content in the molecule. Inside the same series of the prepared compounds, the cloud points are related to their HLB values; it increases with increasing in HLB values.

#### 4.1.3 Wetting time

All prepared nonionic surfactants have good wetting time. From the data in Tables 4, 5 and 6 it can be seen that the wetting time decreases with increasing ethylene oxide units in the molecule. Also the type 1 shows highest wetting time and type 3 shows the lowest one.

Table 5:

Surface properties of synthesized succinate mixed esters with alkyl and polyglycol groups ( $\text{Na}_2\text{CO}_3$ ) (Type 2.)  $\text{ROOCCH}_2\text{CH}_2\text{COO}(\text{CH}_2\text{CH}_2\text{O})_n\text{H}$

Comp.	n	Surface Tension (dyne/cm) 0.1 wt%	Interfacial tension (dyne/cm) 0.1 wt%	Cloud Point °C 1.0 wt%	Wetting time (sec.) 0.1 wt%	Emulsion stability (min:sec) 20m.mole	Foam height (mm) 0.1 wt%	Calc. HLB
<b>C8 a</b>	3	27.0	7.0	50	40	70:00	55	12.18
<b>b</b>	5	27.5	7.5	53	38	75:00	60	13.71
<b>c</b>	7	28.0	8.0	55	35	75:00	65	14.74
<b>d</b>	10	29.0	8.5	60	32	82:38	68	15.78
<b>C10 a</b>	3	28.0	8.0	52	45	74:00	60	11.31
<b>b</b>	5	28.5	8.5	55	43	75:25	62	12.91
<b>c</b>	7	29.0	9.0	58	42	78:00	69	14.01
<b>d</b>	10	30.0	10.0	62	40	84:50	75	15.15
<b>C12 a</b>	3	28.5	9.0	55	50	70:25	65	10.55
<b>b</b>	5	29.0	10.0	60	48	72:00	70	12.19
<b>c</b>	7	30.0	10.5	62	45	73:42	75	13.35
<b>d</b>	10	31.0	11.0	65	43	75:00	80	14.36
<b>C14 a</b>	3	29.0	10.0	56	55	60:15	70	9.88
<b>b</b>	5	29.5	10.5	62	53	62:00	75	11.56
<b>c</b>	7	30.0	11.0	65	50	65:45	80	12.75
<b>d</b>	10	32.0	12.0	68	46	70:20	86	14.03
<b>C16 a</b>	3	30.0	11.0	64	60	52:15	75	9.30
<b>b</b>	5	30.5	11.5	65	58	55:14	80	10.98
<b>c</b>	7	31.0	12.0	68	55	58:00	85	12.20
<b>d</b>	10	33.0	12.5	72	52	60:31	88	13.52
<b>C18 a</b>	3	30.0	12.0	65	60	45:49	78	8.78
<b>b</b>	5	31.0	12.5	70	63	48:00	80	10.49
<b>c</b>	7	32.0	13.0	73	60	50:00	88	11.69
<b>d</b>	10	34.0	13.5	78	56	55:38	95	13.05

Table 6:

Surface properties of synthesized ethoxylated succinic half ester ethanol amides (k10 clay) (Type3)  $\text{ROOCCH}_2\text{CH}_2\text{CON}(\text{CH}_2\text{CH}_2\text{O})_n+1\text{H}$

Comp.	n	Surface Tension (dyne/cm) 0.1 wt%	Interfacial tension (dyne/cm) 0.1 wt%	Cloud Point °C 1.0 wt%	Wetting time (sec.) 0.1 wt%	Emulsion stability (min:sec) 20m.mole	Foam height (mm) 0.1 wt%	Calc. HLB
<b>C8 a</b>	3	25.0	7.0	55	35	73:38	53	12.45
<b>b</b>	5	26.0	8.0	60	32	75:55	55	13.69
<b>c</b>	7	26.5	8.0	65	30	78:00	58	14.58
<b>d</b>	10	28.0	8.5	70	28	85:00	63	15.53
<b>C10 a</b>	3	26.0	8.0	55	40	70:00	55	11.72
<b>b</b>	5	27.5	8.5	60	38	72:15	58	13.00
<b>c</b>	7	28.0	9.0	70	35	75:00	65	13.95
<b>d</b>	10	29.0	9.5	75	32	77:20	67	14.95
<b>C12 a</b>	3	27.0	9.0	65	45	68:55	60	11.42
<b>b</b>	5	28.0	10.0	66	42	70:00	65	12.94
<b>c</b>	7	29.0	10.5	70	40	73:00	70	13.01
<b>d</b>	10	30.0	11.0	75	38	74:00	72	14.06
<b>C14 a</b>	3	28.0	10.0	66	50	65:00	65	11.07
<b>b</b>	5	28.5	10.0	68	48	63:00	66	12.38
<b>c</b>	7	29.0	10.5	72	48	65:00	70	13.38
<b>d</b>	10	31.5	11.0	78	45	68:46	75	14.46
<b>C16 a</b>	3	29.0	10.5	68	55	60:35	70	10.48
<b>b</b>	5	29.5	11.0	70	52	62:44	73	11.84
<b>c</b>	7	30.0	11.0	74	50	66:00	76	12.85
<b>d</b>	10	32.0	11.5	80	50	69:30	80	13.48
<b>C18 a</b>	3	30.0	11.0	70	58	58:00	73	9.96
<b>b</b>	5	31.0	11.5	75	53	60:00	75	11.33
<b>c</b>	7	32.5	12.0	80	50	64:00	78	12.36
<b>d</b>	10	33.0	12.5	83	47	69:55	84	13.52

#### 4.1.4 Emulsion stability

The emulsion stability of the prepared nonionic surfactants was determined by standard methods (Tables 4–6) [17]. All prepared nonionic surfactants possess good emulsion stability. The emulsion stability increases with increasing hydrophobic and hydrophilic chain length. Also it appears that the type 3 has highest emulsification stability.

#### 4.1.5 Foam height

The foaming of the prepared nonionic compounds was investigated. The measured data are given in Tables 4–6. It can be seen that the nonionic surfactants prepared have low foam-height as other of nonionics [27]. Also the foam-height increases with increasing number of ethylene oxide units rather than with the length of hydrophobic part.

Table 7:

Biodegradability of the prepared succinic acid base surfactants, type 1.

Comp.	No.	1 <sup>st</sup> day	2 <sup>nd</sup> day	3 <sup>rd</sup> day	4 <sup>th</sup> day	5 <sup>th</sup> day	6 <sup>th</sup> day	7 <sup>th</sup> day
<b>C8 a</b>	3	63	68	75	82	90	98	–
<b>b</b>	5	62	65	70	75	85	91	98
<b>c</b>	7	60	64	68	74	82	88	95
<b>d</b>	10	59	62	67	73	80	85	95
<b>C10 a</b>	3	62	69	73	79	86	94	99
<b>b</b>	5	58	64	69	73	80	88	96
<b>c</b>	7	56	61	65	69	79	85	94
<b>d</b>	10	55	60	64	67	77	82	94
<b>C12 a</b>	3	58	64	72	79	85	92	98
<b>b</b>	5	57	60	66	72	83	88	95
<b>c</b>	7	54	58	65	70	82	88	94
<b>d</b>	10	53	55	60	68	75	82	93
<b>C14 a</b>	3	55	64	71	78	83	90	97
<b>b</b>	5	54	57	65	71	80	89	95
<b>c</b>	7	52	55	62	68	78	87	93
<b>d</b>	10	51	53	60	66	74	80	92
<b>C16 a</b>	3	53	64	72	77	82	89	96
<b>b</b>	5	52	56	64	69	78	86	94
<b>c</b>	7	51	54	63	67	75	84	93
<b>d</b>	10	50	53	60	66	73	80	90
<b>C18 a</b>	3	52	63	71	77	82	88	93
<b>b</b>	5	51	56	61	68	75	85	92
<b>c</b>	7	50	54	60	66	74	83	90
<b>d</b>	10	49	52	59	63	72	80	89

#### 4.1.6 Hydrophilic Lypophilic Balance (HLB)

The HLB values of the prepared nonionic surfactants were calculated using Dieves equations [24]. From the calculated data (given in the Tables 4, 5 and 6) it can be seen that all prepared nonionic surfactant types have HLB ranging from 7.94 to 15.84. These HLB ranges enable the prepared surfactants to be used in many industrial field applications.

#### 4.2 Biodegradability

In the course of degradation the river die-away tests was followed by surface tension measurements [25]. The biodegradability data are given in Tables 7–9. Within the experimental accuracy all prepared nonionic surfactant types seem to degrade easy. Also it can be seen that the type 1 has less degradability than other two types. This may be attributed to the higher hydrophobic content of their molecules. Also the

Table 8:

Biodegradability of the prepared succinic acid base surfactants, type 2.

Comp.	No.	1 <sup>st</sup> day	2 <sup>nd</sup> day	3 <sup>rd</sup> day	4 <sup>th</sup> day	5 <sup>th</sup> day	6 <sup>th</sup> day	7 <sup>th</sup> day
<b>C8 a</b>	3	65	71	79	86	92	99	–
<b>b</b>	5	63	70	78	84	90	99	–
<b>c</b>	7	62	69	76	82	89	97	–
<b>d</b>	10	61	66	75	81	86	95	–
<b>C10 a</b>	3	63	70	79	85	91	99	–
<b>b</b>	5	62	68	77	84	90	98	–
<b>c</b>	7	60	67	76	83	89	97	–
<b>d</b>	10	59	66	74	80	88	94	–
<b>C12 a</b>	3	62	70	78	84	90	99	–
<b>b</b>	5	61	69	76	83	89	98	–
<b>c</b>	7	60	67	75	82	88	95	–
<b>d</b>	10	59	65	74	81	87	93	–
<b>C14 a</b>	3	60	68	77	82	88	96	–
<b>b</b>	5	59	66	75	81	86	95	–
<b>c</b>	7	57	65	74	80	85	93	99
<b>d</b>	10	56	64	73	79	83	92	99
<b>C16 a</b>	3	58	66	75	81	87	96	–
<b>b</b>	5	57	65	74	79	86	94	–
<b>c</b>	7	55	63	72	78	84	92	99
<b>d</b>	10	54	62	71	77	82	90	99
<b>C18 a</b>	3	56	65	74	80	86	94	99
<b>b</b>	5	55	62	73	78	85	92	99
<b>c</b>	7	53	61	71	76	84	91	98
<b>d</b>	10	52	60	69	75	81	88	96

biodegradability rate decreased with increasing number of the carbon atoms in the hydrophobic part. This led to conclusion that a longer hydrophobic part makes the diffusion of the molecule through the cell membrane and their degradation more difficult.

Table 9:

Biodegradability of the prepared succinic acid base surfactants, type 3.

Comp.	No.	1 <sup>st</sup> day	2 <sup>nd</sup> day	3 <sup>rd</sup> day	4 <sup>th</sup> day	5 <sup>th</sup> day	6 <sup>th</sup> day	7 <sup>th</sup> day
<b>C8 a</b>	3	66	72	80	87	93	99	–
<b>b</b>	5	64	71	79	85	91	99	–
<b>c</b>	7	62	69	77	84	90	98	–
<b>d</b>	10	60	68	76	82	88	97	–
<b>C10 a</b>	3	65	71	79	86	92	99	–
<b>b</b>	5	64	69	78	85	91	99	–
<b>c</b>	7	62	68	77	84	90	98	–
<b>d</b>	10	61	67	75	82	89	95	–
<b>C12 a</b>	3	63	72	78	85	91	99	–
<b>b</b>	5	62	71	77	84	90	97	–
<b>c</b>	7	60	69	76	83	89	96	–
<b>d</b>	10	60	68	75	82	88	95	–
<b>C14 a</b>	3	62	71	77	84	90	98	–
<b>b</b>	5	61	70	76	83	89	96	–
<b>c</b>	7	59	69	75	82	88	95	–
<b>d</b>	10	58	67	74	80	86	94	–
<b>C16 a</b>	3	61	70	76	83	89	97	–
<b>b</b>	5	60	69	75	82	88	96	–
<b>c</b>	7	58	67	74	80	87	95	–
<b>d</b>	10	56	66	72	79	86	92	99
<b>C18 a</b>	3	60	68	76	82	87	95	–
<b>b</b>	5	59	67	74	80	86	94	–
<b>c</b>	7	57	6	72	78	84	92	99
<b>d</b>	10	56	64	69	76	82	90	99

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(Kiss Béla)