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# PRODUCING OF ERYTHRITOL, 1,2,3-BUTANETRIOL AND 1,2,4-BUTANETRIOL ESTERS WITH PALMITIC, STEARIC, LAURIC AND OLEIC ACIDS

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New methods for synthesizing polyols have been developed: erythritol, 1,2,3-butanetriol and 1,2,4-butanetriol from 1,4-dichloro-2-butene, 1-chloro-2-butene and 4-chloro-1-butene respectively. The esterification of the resulting erythritol, 1,2,3-butanetriol and 1,2,4-butanetriol with palmitic, stearic, lauric and oleic acids gave the corresponding esters. The physicochemical constants of the obtained compounds are determined. New non-glyceride oil-like products can be used to screen dietary oils and fats.

***Keywords*:** esterification, erythritol, 1,2,3-butanetriol, 1,2,4-butanetriol, stearic, palmitic, lauric and oleic acids.

**Introduction.** Esters of polyhydric alcohols are widely used as lubricating oils, plasticizers, additives to oils, etc. [1]. It is of interest to use sucrose esters of some monosaccharides (mannitol, xylitol, sorbitol, etc.) with higher fatty acids (stearic, palmitic, etc.) as dietary substitutes for oils and fats [2]. Esters of erythritol with higher fatty acids with a degree of substitution of 1 : 1÷2 are used in the food industry as emulsifiers [3, 4]. Individually, erythritol (E 968) is used in food as a substitute for sugar, a stabilizer and a moisturizing agent [5]. The purpose of this investigation is to study the possibility of obtaining esters of erythritol, 1,2,3-butanetriol and 1,2,4-butanetriol with stearic, palmitic, lauric and oleic acids in molar ratios 1 : 2÷4. The resulting compounds may be of interest as dietary substitutes for oils and fats [6]. Screening of new oil and fat substitutes is advisable to carry out with two-, three- and four-substituted esters of fatty acids and polyhydric alcohols of natural origin. These are polyols: sorbitol, mannitol, xylitol, erythritol, as well as fatty acids that are part of natural oils and fats: palmitic, stearic, lauric, oleic, etc.

In our previous publications, data on the production of esters of mannitol, sorbitol, erythritol and 1,4-butanediol with higher fatty acids [7–9], as well as citric acid with higher alcohols ($C_6$–$C_{18}$) are given [10]. In [9] for the preparation of esters of erythritol we used the alkylation reaction of 1,2,3,4-tetrachlorobutane with potassium, sodium and calcium salts of higher carboxylic acids at 140–190°*C* in various solvents. In the present study the results of the synthesis of new non-glyceride

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esters by the esterification of erythritol as well as 1,2,3-butanetriol and 1,2,4-butanetriol by certain higher carboxylic acids in a solution of N-methylpyrrolidone are given. The choice of this solvent is due to its effectiveness in the esterification reaction of erythritol [9].

**Experimental Part.**

***Characteristics of Synthesized Compounds.*** The individuality of the synthesized esters is confirmed by the NMR and IR spectra (Figs. 1, 2). Vibrational bands of erythritol oscillations are observed in the range of 3000–3500 $cm^{-1}$. Vibrational bands of vibrations of carboxylic acids are observed in the range 1740 and 3000–3600 $cm^{-1}$. After obtaining the esterification product – erythritol tetrapalmitate, the bands of hydroxyl groups disappear, and the band at 1740 $cm^{-1}$ persists. This result indicates that the reaction between erythritol and fatty acid is complete, and all the hydroxyl groups of erythritol and the carboxyl groups of the carboxylic acids are converted to ester bonds. The peaks of the carbonyl groups of the tetrasubstituted esters formed confirm the esterification reaction. In the case of the preparation of trisubstituted and disubstituted esters of erythritol, disubstituted, trisubstituted and tetrasubstituted products are simultaneously present in the reaction mixture. Similarly, the reactions of 1,2,3-butanetriol and 1,2,4-butanetriol with carboxylic acids proceed.

***Synthesis of the Starting Polyols.*** 1-Chloro-2-butene was used for the synthesis of 1,2,3-butanetriol. 1-Chloro-2-butene is a by-product of the production of chloroprene at the chemical plant of CJSC "Nairit" [11]. Hydrolysis of monochlorobutene gave 1-hydroxy-2-butylene (crotyl alcohol) with an admixture of 2-hydroxy-3-butene in a ratio of approximately 3 : 1. The reaction of crotyl alcohol with hydrogen peroxide and acetic anhydride gave 1-hydroxy-2,3-epoxybutane, which was heated with sodium carbonate to give 1,2,3-butanetriol (Scheme 1). Accordingly, epoxidation and hydrolysis of 2-hydroxy-3-butene result in identical 1,2,3-butanetriol.

$$\mathrm{ClCH_2CH=CHCH_3} \xrightarrow{\mathrm{HCOONa/H_2O}} \mathrm{HOCH_2CH=CHCH_3 + CH_2=CHCH(OH)CH_3},$$

$$\mathrm{HOCH_2CH=CHCH_3} \xrightarrow{\mathrm{H_2O_2+(CH_3CO)_2O+H_2O}} \mathrm{HOCH_2CH(OH)CH(OH)CH_3}, \qquad (1)$$

$$\mathrm{CH_2=CHCH(OH)CH_3} \xrightarrow{\mathrm{H_2O_2+(CH_3CO)_2O+H_2O}} \mathrm{HOCH_2CH(OH)CH(OH)CH_3}.$$

4-Chloro-1-butene, which is also a by-product of the synthesis of chloroprene [11], was used for the synthesis of 1,2,4-butanetriol. Hydrolysis of monochlorobutene gave 1-hydroxy-3-butylene. Its oxidation with hydrogen peroxide with acetic anhydride gave 1-hydroxy-3,4-epoxybutane, which was heated with sodium carbonate to give 1,2,4-butanetriol (Scheme 2).

$$\mathrm{2CH_2=CHCH_2CH_2Cl} \xrightarrow{\mathrm{HCOONa/H_2O}} \mathrm{2CH_2=CHCH_2CH_2OH},$$

$$\mathrm{CH_2=CHCH_2CH_2OH} \xrightarrow{\mathrm{H_2O_2+(CH_3CO)_2O+H_2O}} \mathrm{HOCH_2CH(OH)CH_2CH_2OH}. \qquad (2)$$

For the synthesis of erythritol was used 1,4-dichloro-2-butene, which is an intermediate and a by-product of the synthesis of 2-chlorobutadiene [11]. Hydrolysis of 1,4-dichloro-2-butene gave 1,4-dihydroxy-2-butene with an admixture

of 1,2-dihydroxy-3-butene [12]. Reaction of mixture of 2-butene-1,4-diol and 3-butene-1,2-diol with hydrogen peroxide and acetic anhydride gave a mixture of 1,4-dihydroxy-2,3-epoxybutane with 1,2-dihydroxy-3,4-epoxybutane [13]. Heating of the epoxy compounds with sodium carbonate solution gave a mixture of meso- and d, l-stereoisomers of 1,2,3,4-tetrahydroxybutane (erythritol) (Scheme 3).

$$ClCH_2CH=CHCH_2Cl \xrightarrow{HCOONa/H_2O} HOCH_2CH=CHCH_2OH + HOCH_2CH(OH)CH=CH_2,$$

$$HOCH_2CH=CHCH_2OH \xrightarrow{H_2O_2+(CH_3CO)_2O+H_2O} HOCH_2CH(OH)CH(OH)CH_2OH, \quad (3)$$

$$HOCH_2CH(OH)CH=CH_2 \xrightarrow{H_2O_2+(CH_3CO)_2O+H_2O} HOCH_2CH(OH)CH(OH)CH_2OH.$$

Experimental data on the synthesis of polyols are summarized in Tabs. 1, 2.

*Table 1*

*Hydrolysis of monochlorobutenes in an aqueous solution at 100°C*

| Monochlorobutene | Salt of carbonic acid | Molar ratio of reagents | Concent-ration of salt, % | Reaction duration, *h* | Yields of glycols, mol. % | |
|---|---|---|---|---|---|---|
| 1-chloro-2-butene | $CH_3COONa$ | 1 : 1.1 | 10 | 3 | 2-butene-1-ol, 48 | 3-butene-2-ol, 24 |
| 1-chloro-2-butene | $CH_3COONa$ | 1 : 2.0 | 20 | 2 | 2-butene-1-ol, 42 | 3-butene-2-ol, 25 |
| 1-chloro-2-butene | HCOONa | 1 : 1.1 | 10 | 3 | 2-butene-1-ol, 46 | 3-butene-2-ol, 30 |
| 1-chloro-2-butene | HCOONa | 1 : 2.0 | 25 | 4 | 2-butene-1-ol, 43 | 3-butene-2-ol, 25 |
| 1-chloro-2-butene | $(COONa)_2$ | 1 : 1.1 | 10 | 3 | 2-butene-1-ol, 25 | 3-butene-2-ol, 22 |
| 1-chloro-2-butene | $(COONa)_2$ | 1 : 2.0 | 30 | 5 | 2-butene-1-ol, 45 | 3-butene-2-ol, 31 |
| 4-chloro-1-butene | $CH_3COONa$ | 1 : 2.0 | 20 | 3 | 3-butene-1-ol, 61 | – |
| 4-chloro-1-butene | HCOONa | 1 : 2.0 | 20 | 3 | 3-butene-1-ol, 55 | – |
| 4-chloro-1-butene | $(COONa)_2$ | 1 : 2.0 | 20 | 3 | 3-butene-1-ol, 53 | – |

*Table 2*

*Oxidation of 2-butene-1,4-diol, mixture of 2-butenol-1 and 3-butenol, and 3-butene-1-ol with hydrogen peroxide with acetic anhydride and subsequent hydration in situ with 10% sodium carbonate solution*

| Unsaturated alcohol | Concentra-tion of $H_2O_2$, % | Molar ratio of butenol, $H_2O_2$ and $(CH_3CO)_2O$ | Temperature, °*C* | Reaction duration, *h* | Yields of alcohols, mol. % |
|---|---|---|---|---|---|
| 2-butene-1,4-diol | 30 | 1 : 1.1 : 1.1 | 20 | 4 | erythritol , 60 |
| –"– | 30 | 1 : 2 : 2 | 20 | 4 | –"– , 75 |
| –"– | 30 | 1 : 3 : 3 | 20 | 4 | –"– , 81 |
| –"– | 60 | 1 : 2 : 2 | 20 | 4 | –"– , 76 |
| –"– | 30 | 1 : 2 : 2 | 0 | 6 | –"– , 66 |
| –"– | 60 | 1 : 2 : 0 | 0 | 3 | –"– , 63 |
| 2-butene-1-ol and 3-butene-2-ol | 30 | 1 : 2 : 2 | 20 | 4 | 1,2,3-butanetriol, 80 |
| –"– | 30 | 1 : 2 : 2 | 0 | 4 | –"– , 73 |
| 3-butene-1-ol | 30 | 1 : 2 : 2 | 20 | 4 | 1,2,4-butanetriol, 75 |

***The Esterification of Erythritol, 1,2,3-Butanetriol and 1,2,4-Butanetriol with Stearic, Palmitic, Lauric and Oleic Acids.*** The esterification reaction of the resulting polyols is carried out in a conventional manner on a combined reactive distillation unit, without the use of acid catalysts. During the reaction, dispersion of the polyols and acids in solvent is essential. The process is carried out at a temperature of 120–220°*C* with the distillation of water vapor (4)–(6) liberated during the reaction. The use of N-methylpyrrolidone solvent is advisable to ensure homogeneity of the reaction mixture, since reagents do not mix in this temperature range.

$$HOCH_2CH(OH)CH(OH)CH_3 + 3R^1COOH \rightarrow \rightarrow R^1COOCH_2CH(OOCR^1)CH(OOCR_1)CH_3, \quad (4)$$

$$HOCH_2CH(OH)CH_2CH_2OH + 3R^1COOH \rightarrow \rightarrow R^1COOCH_2CH(OOCR^1)\ CH_2CH_2OOCR^1, \quad (5)$$

$$HOCH_2CH(OH)CH(OH)CH_2OH + 4R^1COOH \rightarrow \rightarrow R^1COOCH_2CH(OOCR^1)CH(OOCR_1)CH_2OOCR^1. \quad (6)$$

*Table 3*

*The esterification of erythritol by higher carboxylic acids in a solution of N-methylpyrrolidone*

| № | Acid | Ratio of polyol to acid | Temperature, °*C* | Duration of reaction, *h* | Yield, mol. % | Freez. point, °*C*, physical property |
|---|---|---|---|---|---|---|
| 1 | palmitic | 1 : 2 | 160 | 8 + 2 | 97 | 57–59, soft |
| 2 | palmitic | 1 : 3 | 160 | 8 + 2 | 96 | 58–62, soft |
| 3 | palmitic | 1 : 4 | 160 | 8 + 2 | 93 | 97–100, hard |
| 4 | palmitic | 1 : 5 | 160 | 8 + 2 | 96 | 100–102, hard |
| 5 | palmitic | 1 : 4 | 190 | 8 + 2 | 92 | 90–101, hard |
| 6 | palmitic | 1 : 4 | 220 | 8 + 2 | 92 | 90–106, hard burnt |
| 7 | palmitic | 1 : 4 | 140 | 8 + 2 | 68 | 90–106, hard |
| 8 | palmitic | 1 : 4 | 120 | 8 + 2 | 32 | 90–106, hard |
| 9 | palmitic | 1 : 4 | 160 | 6 + 2 | 32 | 90–106, hard |
| 10 | palmitic | 1 : 4 | 160 | 16 + 2 | 96 | 90–103, hard |
| 11 | palmitic | 1 : 4 | 160 | 20 + 2 | 96 | 90–103, hard burnt |
| 12 | stearic | 1 : 2 | 160 | 8 + 2 | 96 | 47–48, hard |
| 13 | stearic | 1 : 3 | 160 | 8 + 2 | 96 | 90–96, hard |
| 14 | stearic | 1 : 4 | 160 | 8 + 2 | 97 | 98–102, hard |
| 15 | lauric | 1 : 2 | 160 | 8 + 2 | 97 | 47–52, oleaginous |
| 16 | lauric | 1 : 3 | 160 | 8 + 2 | 96 | 70–74, soft |
| 17 | lauric | 1 : 4 | 160 | 8 + 2 | 94 | 88–91, hard |
| 18* | oleic | 1 : 3 | 160 | 8 + 2 | 92 | –29–25, liquid, dark |
| 12* | oleic | 1 : 4 | 160 | 8 + 2 | 94 | –16–10, liquid, dark |
| 13* | stearic + oleic | 1 : 2 : 2 | 160 | 8 + 2 | 90 | 43–45, soft |
| 14* | stearic + oleic | 1 : 1 : 3 | 160 | 8 + 2 | 96 | 30–36, oleaginous |
| 15* | stearic + oleic | 1 : 3 : 1 | 160 | 10 + 2 | 96 | 68–71, soft |
| 16* | palmitic + oleic | 1 : 2 : 2 | 160 | 8 + 2 | 93 | –4–0, honey-like liquid |
| 17* | palmitic + oleic | 1 : 3 : 1 | 160 | 8 + 2 | 95 | 16–20, honey-like |
| 18* | lauric + oleic | 1 : 2 : 2 | 160 | 8 + 2 | 95 | 32, caramelized |
| 19* | lauric + oleic | 1 : 1 : 3 | 160 | 6 | 95 | –10, liquid |
| 20* | lauric + oleic | 1 : 3 : 1 | 190 | 6 (16) | 72 (95) | gelled |

* To inhibit polymerization, P-23 is added.

The reaction time is first monitored by distilling off the water vapor in the condenser then the concentration of the polyols in the reaction mixture is controlled chromatographically. The data are given in Tabs. 3, 4.

Figs. 1 and 2 show the IR spectra of the starting butenoles, carboxylic acids, erythritol and the resulting non-glyceride oils and fats.

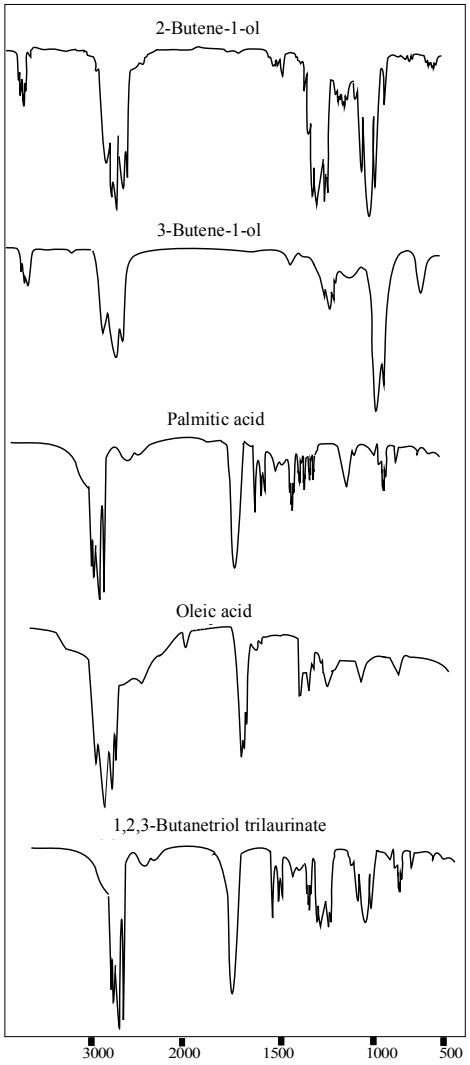


Fig. 1. The IR spectra of 2-butene-1-ol, 3-butene-1-ol, palmic, oleic acids and 1,2,3-butanetriol trilaurinate.

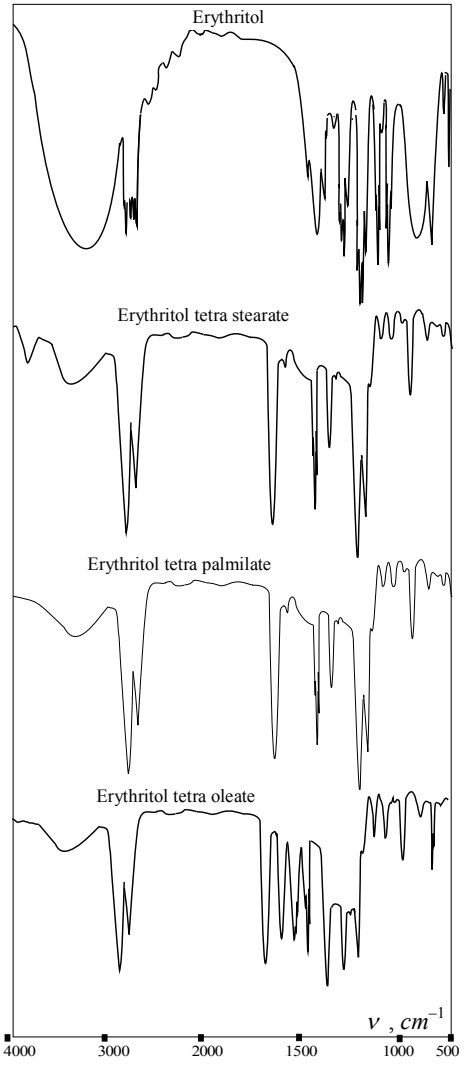


Fig. 2. The IR spectra of erythritol and its esters.

$^1$H NMR spectra were recorded on device Varian Mercury 300 with operating frequency 300 *MHz* in DMSO-$d_6$–$CCl_4$ (1 : 3) with an internal TMS standard. The control of the individuality of the substances is carried out by thin layer chromatography in a toluene + butanol (4 : 1) system on filter paper, iodine vapor used as the developer [14].

IR spectra are obtained on an instrument IRS 29. Spectra of solid samples are taken in vaseline oil on KBr plates in the 4000–400 $cm^{-1}$ region. 1-Chloro-2-butene,

b.p. 84°*C*, has been obtained by rectification of chloroprene production waste (product of the "Nairit" factory in Yerevan), purity is 96.8%. The mixture of isomers: 1-chloro-2-butene and 3-chloro-1-butene was separated on a preparative chromatograph. 4-Chloro-1-butene was isolated by rectification of chloroprene production waste and purified by preparative chromatography, purity 97.1%. Stearic, palmitic, lauric and oleic acids were used standard reagents mark "chemically pure" without further purification.

***Synthesis of Erythritol, 1,2,3-Butanetriol and 1,2,4-Butanetriol.*** Synthesis of erythritol on the basis of 1,4-dichloro-2-butene according to the reaction scheme (6) was carried out. The hydrolysis of 1,4-dichloro-2-butene was carried out according to the procedure of [12]. 1,4-Dihydroxy-2,3-epoxybutane was prepared by epoxidizing 1,4-dihydroxy-2-butene with hydrogen peroxide with acetic anhydride [13]. Erythritol is obtained by hydrolysis of 1,4-dihydroxy-2,3-epoxybutane with 20% aqueous solution of sodium carbonate [12].

Synthesis of 1,2,3-butanetriol and 1,2,4-butanetriol was carried out starting from 1-chloro-2-butene and 4-chloro-1-butene according to reaction schemes (4) and (5), similarly to the synthesis of erythritol. The data are given in Tabs. 1, 2.

*Table 4*

*Esterification of 1,2,3-butanetriol and 1,2,4-butanetriol with higher carboxylic acids in a solution of N-methylpyrrolidone*

| № | Polyol | Acid | Ratio of polyol to acid | *T*, °*C* | Duration of reaction, *h* | Yield, mol. % | Freez. point, °*C*, physical property |
|---|---|---|---|---|---|---|---|
| 1 | 1,2,3-butanetriol | palmitic | 1 : 2 | 160 | 8 + 2 | 97 | 55–58, soft |
| 2 | | palmitic | 1 : 3 | 160 | 8 + 2 | 96 | 68–71, soft |
| 3 | | palmitic | 1 : 4 | 140 | 8 + 2 | 79 | 77–79, hard |
| 4 | | palmitic | 1 : 3 | 190 | 8 + 2 | 92 | 72–76, hard |
| 5 | | palmitic | 1 : 3 | 160 | 16 + 2 | 96 | 80–83, hard |
| 6 | | stearic | 1 : 2 | 160 | 8 + 2 | 95 | 83–86, hard |
| 7 | | stearic | 1 : 3 | 160 | 8 + 2 | 93 | 90–96, hard |
| 8 | | stearic | 1 : 3 | 160 | 16 + 2 | 96 | 92–96, hard |
| 9 | | lauric | 1 : 2 | 160 | 8 + 2 | 97 | 20–25, oleaginous |
| 10 | | lauric | 1 : 3 | 160 | 8 + 2 | 96 | 33–34, oleaginous |
| 11 | | lauric | 1 : 3 | 160 | 16 + 2 | 97 | 34–38, soft |
| 12 | | oleic | 1 : 2 | 160 | 8 + 2 | 94 | –20, liquid |
| 13 | | oleic | 1 : 3 | 160 | 8 + 2 | 92 | –8, liquid |
| 14 | 1,2,4-butanetriol | palmitic | 1 : 2 | 160 | 8 + 2 | 97 | 41–43, soft |
| 15 | | palmitic | 1 : 3 | 160 | 8 + 2 | 96 | 48–50, soft |
| 16 | | palmitic | 1 : 4 | 140 | 8 + 2 | 79 | 46–52, soft |
| 17 | | palmitic | 1 : 3 | 190 | 8 + 2 | 92 | 72–76, soft |
| 18 | | stearic | 1 : 3 | 160 | 8 + 2 | 93 | 82–90, hard |
| 19 | | stearic | 1 : 3 | 160 | 16 + 2 | 96 | 84–90, hard |
| 20 | | lauric | 1 : 2 | 160 | 8 + 2 | 97 | 18–20, oleaginous |
| 21 | | lauric | 1 : 3 | 160 | 8 + 2 | 96 | 31–32, soft |
| 22 | | oleic | 1 : 2 | 160 | 8 + 2 | 94 | –24, liquid |
| 23 | | oleic | 1 : 3 | 160 | 8 + 2 | 92 | –11, liquid |

* To inhibit polymerization, P-23 is added.

***General Procedure for the Esterification.*** The reaction was carried out in the combined reaction-distillation installation comprising a thermostated reactor with a magnetic stirrer and reflux condenser column for condensing the water vapor. The reactor was charged 30 *mL* N-methylpyrrolidone, 2.44 *g* (20 *mmol*) erythritol and 10.25 *g* (40 *mmol*) palmitic acid. With the stirrer on, air was pumped out (10–20 *mm* Hg) and heated to the set temperature. To control the course of the reaction, every 4 *h*, a sample was taken and chromatographically content of starting materials and products have been determined. Then reaction mixture was cooled, the product was washed with water (4×50 *mL*) to remove reagent residues and N-methylpyrrolidone. The resulting oil was recrystallized from 30 *mL* of 80° ethanol and dried to constant weight. The yield of erythritol tetrapalmitate is 22.08 *g* (97%). The reaction product is erythritol tetrapalmitate, which is a soft paraffin-like material of yellow or beige color. The described experiment № 1 and results of the remaining experiments are summarized in Tab. 3. In experiments containing oleic acid, 0.3 *g* of 2,6-di-tert-butyl-4-methylphenol (P-23) was added to the reaction mixture for polymerization inhibition.

Experiments on the esterification of 1,2,3-butanetriol and 1,2,4-butanetriol are carried out similarly. The results of the experiments are given in Tab. 4.

The structures of the obtained erythritol, 1,2,3-butanetriol and 1,2,4-butanetriol esters with palmitic, stearic, lauric and oleic acids were confirmed by IR and $^1$H NMR spectra.

*Erythritol Tetrastearate*. Freez. point 98–102°*C*. IR, *v*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (12H, $CH_3$); 1.18–1.86 m (120 H, $CH_2$); 2.64–2.72 t (8H, $CH_2COO$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*Erythritol Tetrapalmitate*. Freez. point 97–100°*C*. IR, *v*, $cm^{-1}$: 2920, 2850 ($CH_2$); 1738 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (12H, $CH_3$); 1.30 m (88H, $CH_2$); 1.41 m (8H, $CH_2CH_3$); 1.65 m (8H, $CH_2$); 2.30 m (8H, $CH_2COO$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*Erythritol Tetraoleate*. Freez. point –16–10°*C*. IR, *v*, $cm^{-1}$: 3006, 2950, 2920 ($CH_2$); 1671 (C=O); 1645 (CH=); 1463, 1447 ($CH_2$); 1196, 1099 (C–O). $^1$H NMR, *δ*, *ppm*: 0.97 t (12H, $CH_3$); 1.32 t (8H, $COCH_2$); 1.45 m (80H, $CH_2$); 1.60 m (8H, $CH_2$); 2.22 dd (16H, $CH_2CH$=); 2.35 m (8H, CH=); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,3-Butanetriol Tristearate*. Freez. point 85–92°*C*. IR, *v*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (9H, $CH_3$); 1.05 (1H, $CH_3$); 1.31 m (78H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.31 t (6H, $CH_2CO$); 4.08 m (2H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,3-Butanetriol Tripalmitate.* Freez. point 68–71°*C*. IR, *v*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (9H, $CH_3$); 1.05 d (1H, $CH_3$); 1.30 m (66H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.30 t (6H, $CH_2CO$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,3-Butanetriol Trilaurate*. Freez. point 33–38°*C*. IR, *v*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (9H, $CH_3$); 1.05 t (1H, $CH_3$); 1.30 m (42H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.30 t (6H, $CH_2CO$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,4-Butanetriol Tristearate.* Freez. point 84–90°*C*. IR, *ν*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, δ, *ppm*: 0.95 t (9H, $CH_3$); 1.30 m (78H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.30 t (6H, $CH_2CO$); 2.01 t (2H, $CH_2CH_2CH$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,4-Butanetriol Tripalmitate.* Freez. point 48–50°*C*. IR, *ν*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (9H, $CH_3$); 1.30 m ( 66H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.30 t (6H, $CH_2CO$); 2.01 t (2H, $CH_2CH_2CH$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

*1,2,4-Butanetriol Trilaurate.* Freez. point 30–37°*C*. IR, *ν*, $cm^{-1}$: 2919, 2850 ($CH_2$); 1737 (C=O); 1463, 1420 ($CH_2$); 1176, 1132 (C–O). $^1$H NMR, *δ*, *ppm*: 0.95 t (9H, $CH_3$); 1.30 m (42H, $CH_2$); 1.40 m (6H, $CH_2CH_3$); 1.65 m (6H, $CH_2$); 2.30 t (6H, $CH_2CO$); 2.01 t (2H, $CH_2CH_2CH$); 4.08 m (4H, $CH_2O$); 7.25 m (2H, CHCH).

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