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RP-HPLC WITH DETECTION BY MEANS OF ESI-MS AND UV FOR IDENTIFICATION OF CHROMATOGRAPHIC PEAKS OF SOME HYDROXY DERIVATIVES OF EPOXY RESIN

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Abstract. The composition of products synthesized on the basis of ED-24 epoxy resin has been studied using a reversed phase high-performance liquid chromatography followed by an electrospray ionization mass spectrometry (RP-HPLC/ESI-MS). The synthesized products contain free hydroxy groups and such groups, as epoxy, peroxy, carboxy or acrylate.

Keywords: epoxy resin, oligomers with functional groups, RP-HPLC, ESI-MS.

1. Introduction

Epoxy oligomers which are obtained *via* different technologies differ by their composition and content of functional groups. The most often used methods to determine the composition of such compounds are: the high-performance liquid chromatography (HPLC) combined with liquid chromatography at critical conditions (LCCC), size-exclusion chromatography (SEC) and UV-spectroscopic detector at the wave length of 280 nm [1-3]. Individual fractions after chromatographic separation are identified using matrix-assisted laser desorption/ionization (MALDI) with time-of-flight mass spectrometer (MALDI-TOF MS). This method allows to determine the chain-length distribution (CLD) and functionality of the synthesized epoxy oligomers. Combining HPLC and mass spectroscopy is the alternative for bidimensional chromatography which needs two chromatographs. Thermospray mass spectrometry was used by G. Simal *et al.* [4] to identify chromatographic peaks for bisphenol F diglycidyl ether and its hydrolysis products. The mixture of acetonitrile and 0.1 M aqueous solution of ammonium acetate (50:50 v/v) was used as a mobile phase. It is also shown in [4, 5] that apart from liquid chromatorgraphy the gas chromatography with mass spectrometry may be used as well to analyze epoxides based on bisphenol A and F.

V. Dark *et al.* [6] show the possibility to divide epoxy oligomers of glycidyl bisphenol A from DP-O to DP-24 *via* RP-HPLC. They also found bisphenol A, 2,4-bis(a,a-dimethyl-p-hydroxybenzyl)phenol, non-hydrolized chloride-containing compounds and cyclic bis[2,2-is(4-hydroxyphenyl)]propane. However, the authors do not give time axis at chromatograms and do not indicate the rate of eluent composition change.

Van der Maeden *et al.* [7] used gradient HPLC to divide oligomers, namely Epikote DER 671 epoxy resin for oligomers. To find the optimal conditions for RP-HPLC method J. Twitchell *et al.* [8] investigated the eluent composition (tetrahydrofuran, acetonitrile), gradient of mobile phase concentration, eluent rate and column temperature. However they did not identify the peaks but used the obtained results to determine the type of epoxy resin *via* "dactylorgam" method.

Using RP-HPLC the composition of epoxy resin after its extraction from fiber-reinforced composite was determined [9]. To identify the peaks the authors used retention time of the individual components and IR-spectroscopy. The quantitative analysis was carried out using internal standard method. The investigated epoxy resins were found to be a complex mixture of monomers, higher oligomers and by-products. The main components were: tetraglycidyl-4,4'-diamino-diphenyl-methane

(TGDDM), amine hardener – 4,4'-diamino-diphenylsulfone (DDS), monomer – bisphenol A diglycidyl ether (DGEBA).

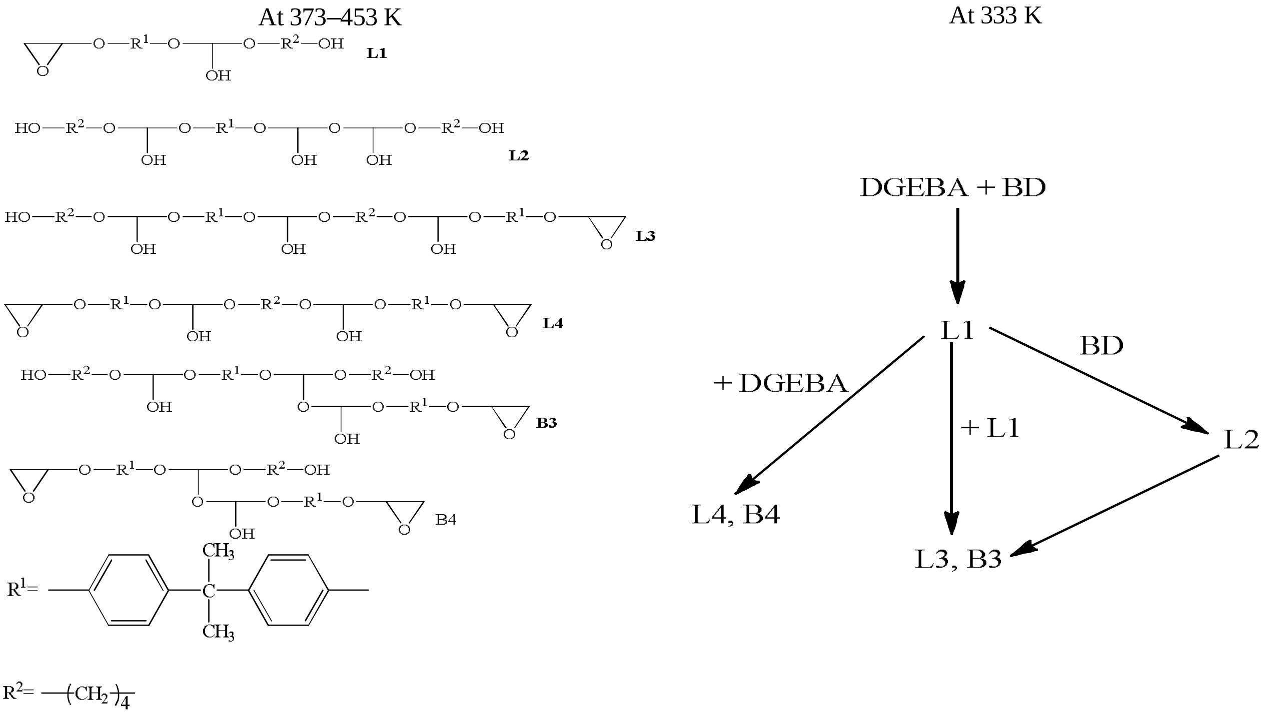
Bisphenols A and F diglycidyl ethers were also determined using HPLC/UV and isocratic eluent – water:methanol:dichlormethane (20:50:30) [10]. To confirm the identity of the investigated compounds the authors propose to use UV-spectrum, obtained by DAD detector. G. Hagnauer and I. Setton [11] used gel-penetrating, normal-phase and HPLC chromatography to analyze the composition of epoxy resins. The oligomeric by-products in bisphenol A diglycidyl ether were studied in [12]. It was found that by-products had not only 2,3-epoxypropyloxy end groups but 2,3-dihydroxypropyloxy, 2-hydroxy-3-metoxypropyloxy or 2-hydroxy-3-para-*tert*-butylphenoxypropyloxy groups as well. Such oligomers were separated and investigated using IR-, NMR- and MS-spectroscopy.

Among modern investigations about using liquid chromatography for separation and identification of epoxy resins the works [13, 14] are of special attention. In [13] the qualitative characteristics of solid epoxy resins were determined using HPLC/ESI-MS. The researchers [14] developed the analytic procedure to study the composition of epoxy and novolac resins blend. They used SEC/MS method based on MALDI-TOF.

S. Shiono *et al.* [12] examined interaction between glycidyl ethers and aliphatic alcohols in the presence of benzyldimethylamine (BDMA) as a catalyst. They suppose the proceeding of reaction between initial epoxide and primary end product and formation of by-products. They confirm the impossibility of branched structures formation during reaction between DGEBA and butanediol in the presence of BDMA at the temperatures higher than 413 K. The possible schemes of products formation are represented in Fig. 1 [15]. The authors also point to the impossibility of using HPLC to analyze the mixtures obtained at 333 K, because at epoxide conversion of above 60 % the gel formation is observed, *i.e.* the molecular mass of the products increases. It should be noted that the values of activation energy obtained in [15] are negative.

Previously [16] we grounded the formation of only linear compounds and low possibility of the reaction between secondary hydroxy group and epoxy one at the temperatures lower than 413 K due to the steric barriers. The values of activation energy were positive, *i.e.* the process rate increases with the increasing temperature. These data conflict with data from [15].

Thus, there are contradictions between researchers concerning the temperature range and the values of activation energy caused by possible difference in used catalytic systems. Of course, this problem needs additional experiments under the same conditions in order to compare the results.


At 373–453 K

At 333 K

Fig. 1. Schemes of products formation during the reaction between alcohols and epoxy compounds at 333 K and within 373–453 K

According to the literature data the gradient mode is most often used to analyze epoxy compounds or their derivatives. While using RP-HPLC the main organic components in an aqueous mobile phase are: tetrahydrofurane [6, 8, 11]; dioxane [7]; acetonitrile [4, 8, 9, 12, 15]; methanol and dichlormethane [10]. It is known [17] that the quality of chromatographic separation decreases with very high or very low values of retention factor. If the retention factor is less than one, the elution ability of mobile phase is too high and it is very difficult to achieve the sufficient separation. So, it is recommended it would be within 1–10.

The possibility of using IR-spectroscopy to confirm the structure of the initial epoxy compounds and butanediol, as well as new synthesized compounds is shown in [18]. NMR spectra were obtained on protons and nuclei of carbon-13 atom [19] that also confirms the structure of the synthesized compounds.

So the aim of this paper is to determine the composition of the synthesized oligomers based on ED-24 epoxy resin using RP-HPLC/ESI-MS/UV method.

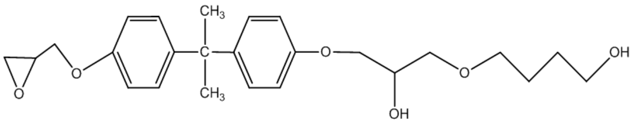
2. Experimental

2.1. Materials

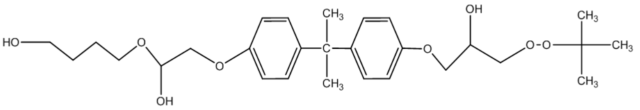
For investigations we used the products obtained *via* synthesis based on ED-24 epoxy resin. The resin was obtained using the procedure described in [18].

The products were:

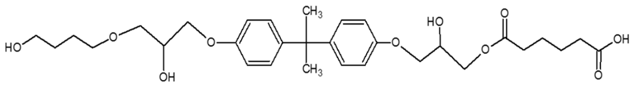
– oligomer with hydroxy and epoxy groups (HDEO)



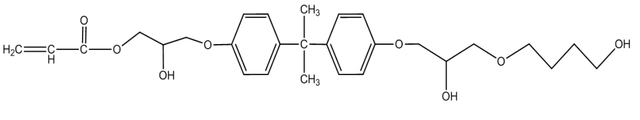
– oligomer with hydroxy and peroxy groups (HPO)



– oligomer with hydroxy and carboxy groups (HCO)



– oligomer with hydroxy and acrylic groups (HAO)



The solutions with concentration of 10 mg in 10 ml of corresponding solvent (100 % methanol or 90 % acetonitrile were used.

2.2. RP-HPLC ESI-MS and UV

Diode array detector (DAD) and mass-spectroscopic (MS) detector were established just after the column. It is the reason we observed the difference in retention time of photometric detector and mass-spectrometer. The retention time of photometric detector is less by 3.6 s (see Fi. 4).

The chromatographic investigations were carried out using the chromatograph (Agilent firm) equipped by the following devices:

– device for sample injection – 1290 Sampler (Wellplate Sampler – G4226A);

– pump for mobile phase feeding – 1290 BinPump (Binary Pump – G4220A);

– thermostat of chromatographic columns – 1290 TCC (Thermostated Column Compartment – G1316C);

– diode array detector for liquid chromatography – 1290 DAD (1290 Infinity Diode Array Detector – G4212A), UV-lamp within the range of 190–400 nm with the step of 2 nm, channel A, B – at 210 and 254 nm, monochromator gap – 4 nm;

– mass spectrometer detector – MS Q-TOF 6540 UHD Accurate Mass Q-TOF LC-MS.

Parameters of chromatographic system

Eluents: methanol – 100 %; acetonitrile:water (v:v) – 90:10 %;

Eluent rates: methanol – 1.5 ml/min; acetonitrile – 0.8 l/min;

Void volume of the column: 1.75 ml;

Zorbax Eclipse XDB column [21] with the length of 150 m and diameter of 4.6 mm*;*

Column temperature – 303 K;

Injection volume – 5 μl;

The final chromatogram was obtained by subtraction the result of blank chromatogram from sample chromatogram under similar conditions of separation and detection. To increase the ionization efficiency the methanol eluent contains sodium ions.

Conditions of ESI-MS operation: acquisition mode MSI: scan range 100–1600 a.m.u.; scan rate – 1.5.

Source parameters: gas temperature 598 K; gas flow – 10 l/min; nebulizer pressure – 35 psi.

Scan source parameters: polarity mode – positive ions; cap voltage – 3500 V; fragmentator – 120 V; skimmer – 45 V; octopole RF peak – 750 V.

Interpretation of mass spectra was carried out according to the general rules [22]. It is known [23] that ions formed under electrospray conditions have less internal energy and their fragmentation does not occur. As a result, mass-spectrum often consists of peaks occurred due to molecular ions with different charges (*e.g.* $[M+nH]^{n+}$). V. Raks *et al.* [24] point that a small number of fragments in mass-spectra is a barrier to form data library for liquid chromatography–mass spectrometry system (LC/MS). Moreover, they studied an effect of eluent molecules (mobile phase) on the formation of ion associates during ionization under atmospheric pressure, ESI ionization in particular. Mass-spectra libraries of other types of ionization are under forming now [24], therefore spectra of new compounds are interpreted according to the general rules using the results of the compounds similar by the structure. All these facts mean that at the present time it is impossible to interpret all masses in spectra within the terms of ion fragments structure.

3. Results and Discussion

At first 100% methanol was used as an eluent and compounds had similar retention time, *i.e.* the peaks were not separated (Fig. 3). However we obtained the initial information about mass-spectral characteristics. The values of retention time and ion mass numbers are represented in Table 1.

In mass spectrum with ESI ionization for butanediol (BD) we observe two main ions with the mass of 113 and 273, corresponding to ion associate of BD with sodium ion (90+23 = 113) and larger associate consisted of two BD molecules and ion fragment of the structure (Fig. 8) suggested by [25], without hydrogen atom (90+90+71–1+23 = 273).

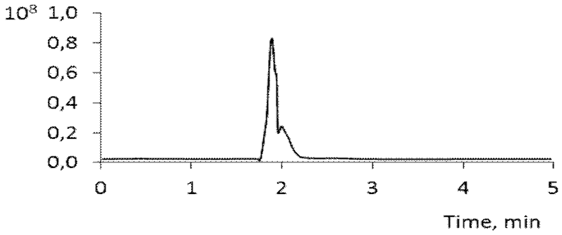


Fig. 3. Total ion current (TIC) chromatogram for HDEO using 100% methanol as the eluent

Table 1

Mass-spectral characteristics of chromatographic peaks for 100% methanol as the eluent

Compound	MS retention time relative to total ionic current (TIC), min	Molecular signals in MS-spectra
BD (*M* = 90.12)	1.21	113. 27
ЕД-24 (*M* = 340.41)	2.024	363. 16
HDEO (*M* = 430.53)	1.912	454.15
HPO (*M* = 520.65)	1.934	471.24
HCO (*M* = 576.28)	1.863	599.28
HAO (*M* = 502.25)	1.856	525.25

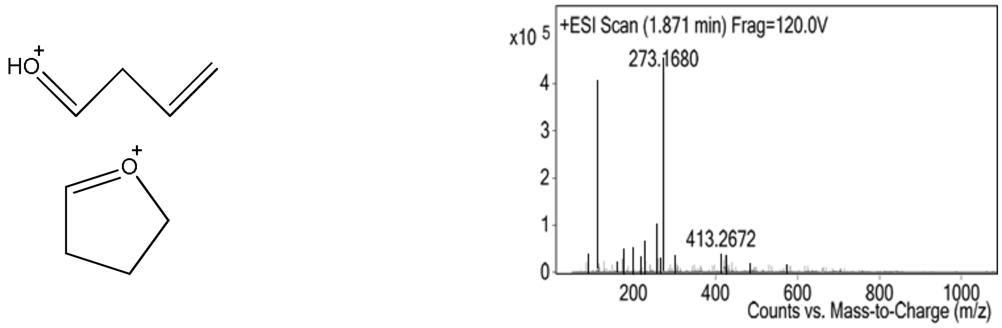


Fig. 4. MS-spectrum of BD using 100% methanol as the eluent and possible structures of ion fragments with the mass of 71

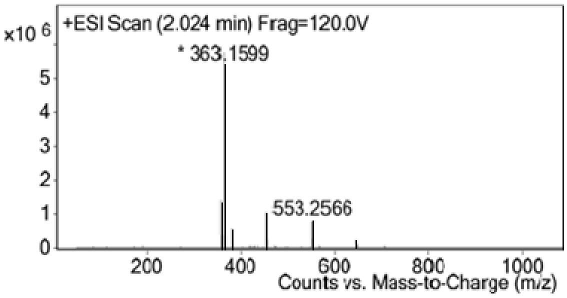
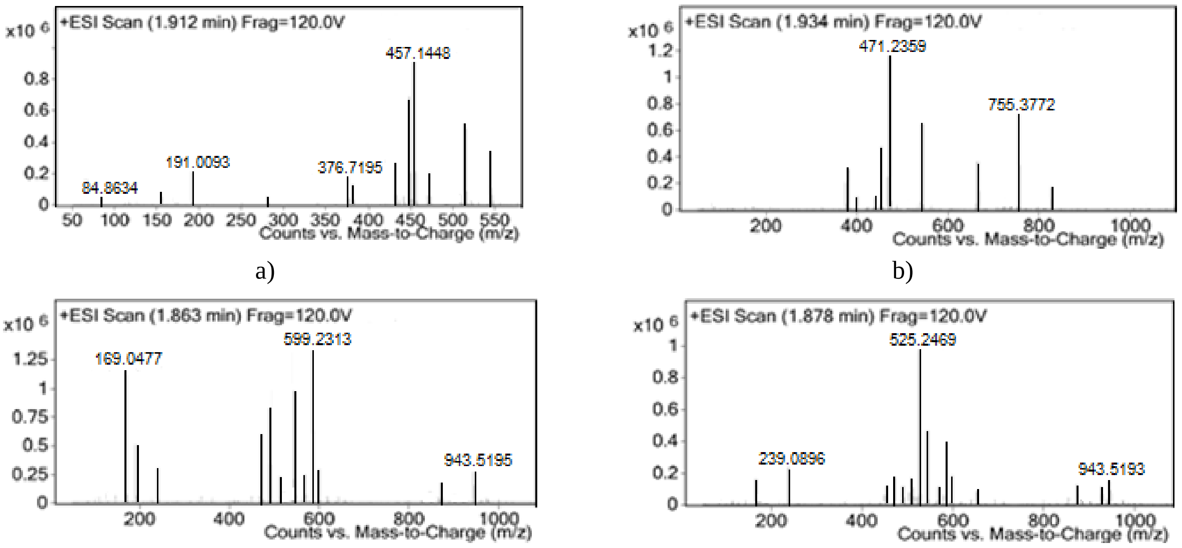


Fig. 5. MS-spectrum of ED-24 using 100% methanol as the eluent



a) b)

c) d)

Fig. 6. MS-spectra of the synthesized compounds using 100 % methanol as the eluent: HDEO (a); HPO (b); HCO (c) and HAO (d)

In mass-spectrum (Fig. 5) with ESI ionization for ED-24 we observe the ion with the smallest mass (*M* = 363), corresponding to ion associate of ED-24 molecule with sodium ion. In mass-spectrum with ESI ionization for HDEO the ion with the mass of 454.15 is interpreted as an ion associate of ED-24 molecule monosubstituted for BD with sodium ion. In mass-spectrum (Fig. 6) with ESI ionization for HPO (retention time is 1.934 min) the ion with *M* = 471.24 is observed, that is interpreted as the ion associate of molecular fragment without tertbutyloxy-group for BD monosubstituted ED-24 resin with the sodium ion. For HCO (retention time is 1.863 min) the ion with *M* = 525.25 is interpreted as the ion associate of molecular fragment with the sodium ion.

According to trial chromatography (100% methanol as the eluent) we determined the main signals in MS corresponding to the initial compounds and reaction products. Retention times of the reaction products are similar (Table 2). The separation for this strong eluent is bad (Fig. 3).

According to the literature data acetonitrile is the most often used eluent for RP- HPLC of epoxy resins because this organic compound increases elution ability of the mobile phase. Gradient elution provides long chromatography (30–60 min) necessary for complex oligomeric mixtures. Among investigated compounds in the present work, ED-24 is the most hydrophobic one without hydroxy or carboxy groups. Retention time for ED-24 on 100% methanol is 2.024 min, corresponding to the capacity factor of (2.00–1.17)/1.17 = 0.71. While transferring to the mobile phase containing acetonitrile at first approximation we may use the ratio for isoeluotropic mobile phases [17] while transferring from methanol $j\,(MeCN) = 0.78 \cdot j\,(MeOH)$. So, to achieve the same strength of the eluent it is enough to use 80 vol % of acetonitrile, but it should be noted that this ratio valid only till the middle of the concentration range. We can also use the simple rule – with the increase in organic modifier concentration by 10 vol % the retention factor decreases by 2-3 times. This rule approximately represents the straight dependence of retention factor logarithm on the volumetric part (φ) of organic modifier in the mobile phase $\ln k = \ln k_0 + S \cdot j$ [26, 27]. Let us assume that with the increase of acetonitrile concentration to 90 % the retention factor increases by three times. If we take this value as a basis for 100% methanol, then 0.71·3 = 2.3. The obtained value is in agreement with the results for bisphenol A diglycidyl ether (DGEBA) [9]. Retention times at different columns are 10.17 min (*k* = (10.17–2.9)/2.9 = 2.51) and 14.04 min (*k* = (14.04–3.49)/3.49 = 3.02). Eluent composition at the moment of peak output was 84 and 97 vol %, respectively. Thus, to obtain the retention factor within the recommended range [17] we may use acetonitrile eluent with water content of 10 vol %.

Isocratic mode of chromatography with 90 % acetonitrile as the eluent allows to improve separation and increase retention time. For instance, retention time for ED-24 increases from 2.024 to 12.821 min, *i.e.* retention factor is (12.82–2.19)/2.19 = 4.85, corresponding to the middle of the recommended range [17].

TIC chromatograms for BD and ED-24 resin are presented in Figs. 7 and 8b. Retention time is 2.24 min for BD and 12.82 min – for ED-24. One can see that actually BD is not held on the column for this strong eluent because it contains two hydrophilic hydroxy groups. Its retention time is somewhat higher than dead time of the system (2.19 min). On DAD chromatogram there is no peak within this range of times because BD absorbs UV-radiation at 210 nm badly. On TIC chromatograms the BD peak is also absent because we investigated not reaction mixtures but the reaction products after washing out of the catalyst and BD.

During chromatographic analysis the UV-detector operates at 210 and 254 nm. On both chromatograms we observe the peak with retention time within 12.760–12.773 min. For the chromatographic peak with retention time of 12.760 min (Fig. 8a) we obtained UV-spectrum represented in Fig. 9. This spectrum has the absorption maxima: large maximum at 225 nm and small wide one – at 280 nm corresponding to the aromatic structure in ED-24 resin. All results are in agreement with literature data [10] – maxima at 225 and 275 nm.

TIC chromatograms for the synthesized compounds are represented in Fig. 10. For MS-detector the values of retention time and mass numbers of ions using 90 % acetonitrile as the eluent are given in Table 2.

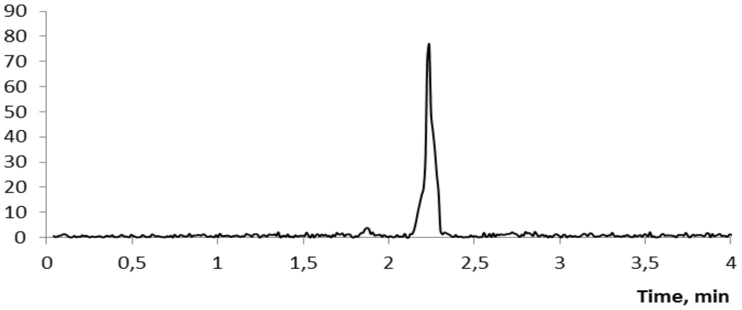


Fig. 7. TIC chromatogram for BD using 90% acetonitrile as the eluent

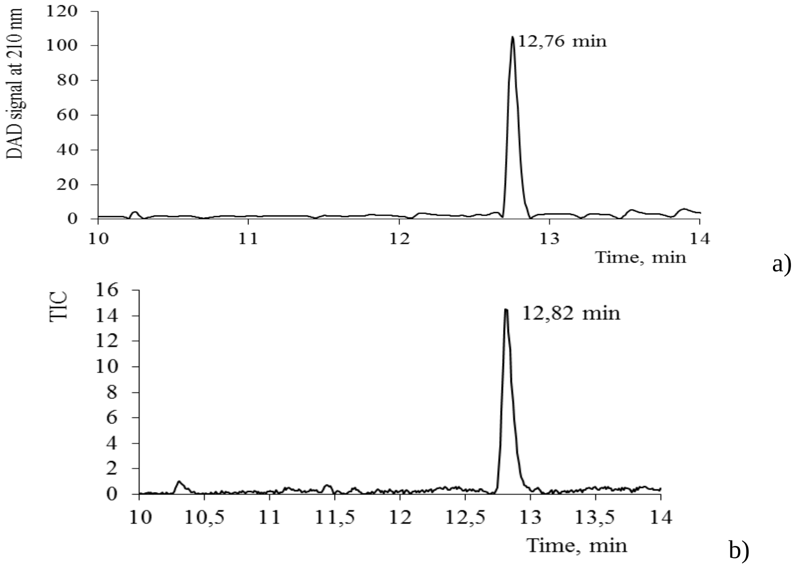


Fig. 8. Comparison of chromatograms for ED-24 obtained from two sequence detectors located after column: DAD (a) and MS (b)

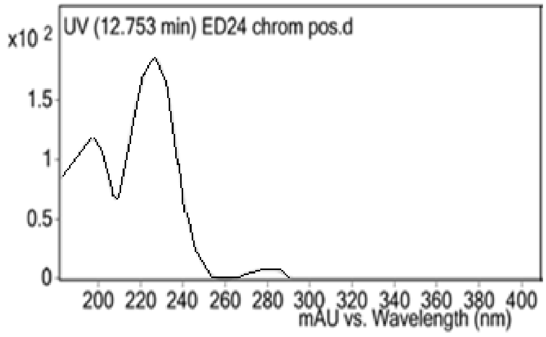
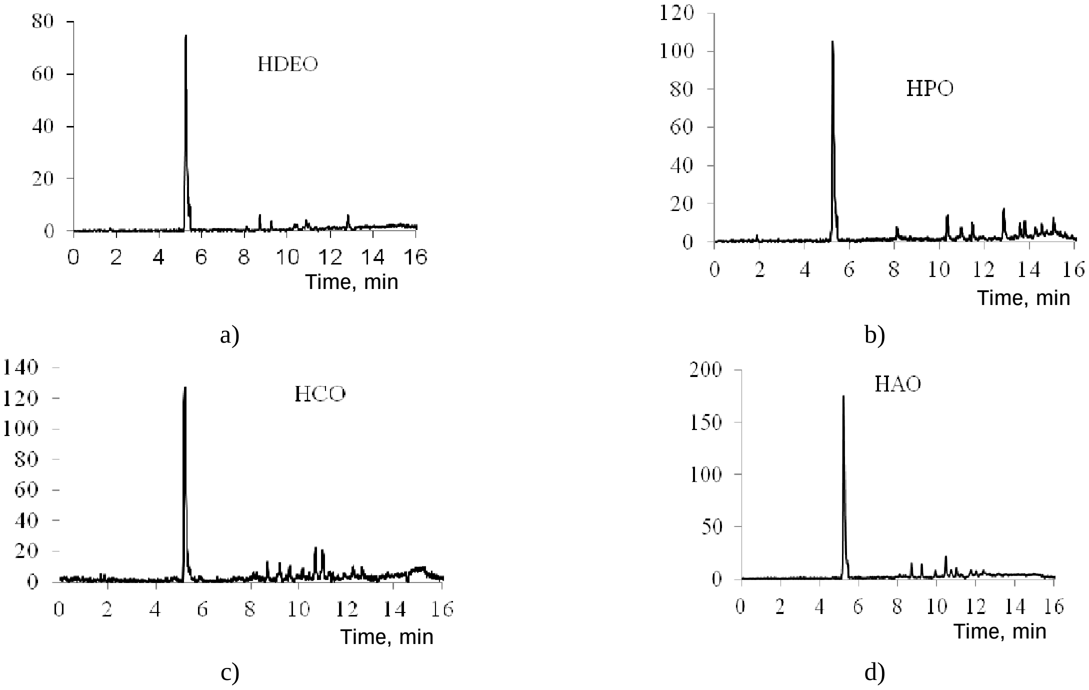


Fig. 9. UV-spectrum of ED-24 chromatographic peak using 90 % acetonitrile as the eluent



a) b)

c) d)

Fig. 10. TIC chromatograms for the synthesized compounds using 90 % acetonitrile as the eluent: HDEO (a); HPO (b); HCO (c) and HAO (d)

Table 2

Mass-spectral characteristics of chromatographic peaks for 90 % acetonitrile as the eluent

Compound	MS retention time relative to total ionic current (TIC), min	Molecular signals in MS-spectra
BD (*M* = 90.12)	2.192	107.1654 130.1346
ЕД-24 (*M* = 340.41)	12.821	357.1927 379.1304 380.1328
HDEO (*M* = 430.53)	5.257	491.2994
HPO (*M* = 520.65)	5.254	486.2914
HCO (*M* = 576.28)	5.215	593.2037
HAO (*M* = 502.25)	5.225	542.1669

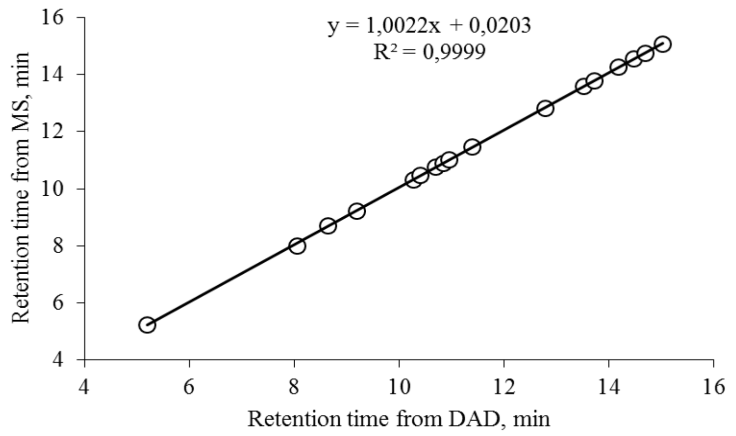


Fig. 11. Ratio between peak times for MS- and DAD-detectors

The peak with retention time of 12.821 min is observed at TIC chromatogram of ED-24 (Fig. 8b). In mass-spectrum of this compound we observe the signals differing from methanol eluent due to the content of water (10 %) and acetonitrile (90 %) in the eluent. There are three main signals in MS-ESI spectrum: i) the ion with the smallest mass of 357.1927 corresponding to ion associate of ED-24 molecule with water – [M+H_2O-H]; ii) signal 379.1304 – [M+can-2H]; iii) signal 380.1328 – [M+AcN-H].

TIC chromatogram for HDEO shows the peak with retention time of 5.234 min. In such a case MS/ESI spectrum has an intensive signal with the mass of 491.2994 corresponding to ion associate of HDEO molecule with the ion of water and acetonitrile molecule [M+AcN+H_2O].

The ratio between peak times for MS- and DAD-detectors is given in Fig. 11. The ratio is well approximated by a straight line with slope coefficient close to 1 and interval of 0.02 min (1.2 s) that corresponds to the difference of 0.05 min between retention times for ED-24 peaks at both detectors. According to Fig. 11 all peaks of impurities appear at chromatograms of both MS- and DAD-detectors at 210 nm, and so these impurities contain aromatic fragment, which is a part of ED-24 structure. The ratio between components may be approximately estimated relative to the signal of spectrophotometric detector at the length of light wave corresponding to the absorption of aromatic fragment in the synthesized compounds using the area of chromatographic peaks as a primary parameter.

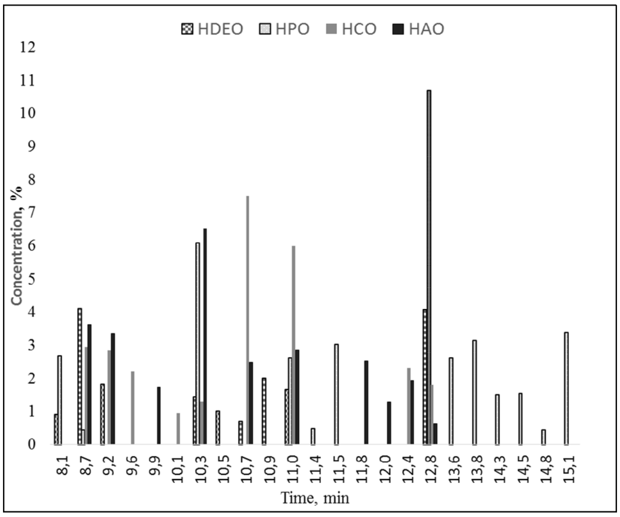


Fig. 12. Concentration of impurities in the synthesized compounds according to the retention times (using 90% acetonitrile as the eluent)

Table 3

Peak areas of TIC chromatograms using 90 % acetonitrile as the eluent

HDEO		HPO		HCO		HAO	
Time, min	Peak areas, %	Time, min	Peak areas, %	Time, min	Peak areas, %	Time, min	Peak areas, %
5.245	82.31	5.220	61.40	5.231	72.11	5.225	73.01
8.102	0.91	8.132	2.67	–	–	–	–
8.698	4.11	8.694	0.43	8.694	2.96	8.711	3.64
9.227	1.82	–	–	9.224	2.85	9.240	3.35
–	–	–	–	9.632	2.22	–	–
–	–	–	–	–	–	9.935	1.74
–	–	–	–	10.172	0.96		
10.341	1.43	10.327	6.09	10.448	1.29	10.453	6.53
10.463	1.01	–	–	–	–	–	–
10.771	0.69	–	–	10.702	7.50	10.740	2.48
10.893	1.99	–	–				
11.025	1.66	10.977	2.61	11.01	6.00	11.027	2.86
–	–	11.352	0.48	–	–	–	–
–	–	11.463	3.03	–	–	–	–
–	–	–	–	–	–	11.755	2.52
–	–	–	–	–	–	12.031	1.29
–	–	–	–	12.312	2.33	12.395	1.93
12.821 ED-24	4.07	12.841 ED-24	10.70	12.687 ED-24	1.80	12.704 ED-24	0.64
–	–	13.580	2.60	–	–	–	–
–	–	13.779	3.14	–	–	–	–
–	–	14.253	1.50	–	–	–	–
–	–	14.540	1.54	–	–	–	–
–	–	14.75	0.43	–	–	–	–
–	–	15.069	3.37	–	–	–	–

Moreover, the estimation may be carried out relative to the signal of MS detector under the mode of ions uniquely corresponding to the definite compound. However for MS detection it is necessary to assume that ionization efficiency (relative to formed ions) for different compounds is the same. Peak areas of TIC chromatograms which are normalized to 100 % without taking into account the ionization efficiency are represented in Table 3.

One can see from Fig. 12 that concentration of impurities is within the range of 0.5–11 %. The maximum concentration (10.7 %) of unreacted ED-24 resin is in HPO, and the minimum (0.64 %) – in HAO. Impurities with retention times higher than for ED-24 peak (12.8 min) are typical only for HPO. The reasons are cross-linking ability of peroxy group and formation of compounds with large molecular mass. Moreover, peaks at 11.4 and 11.5 min are observed at HPO chromatogram, whereas they are absent at other chromatograms.

4. Conclusions

The synthesized hydroxy-containing oligomers based on ED-24 epoxy resin were separated using RP-HPLC method under isocratic mode using acetonitrile eluent with 10 % of water.

The main peaks were identified using MS- and DAD-detectors. It was established that peaks of impurities contain aromatic fragment of ED-24.

The composition of impurities for HPO differs from that of other investigated compounds. The formed compounds have retention time higher than that of ED-24 and they probably are condensation products.

The content of the main component in the synthesized compounds was found to be 61–83 % and residual content of ED-24 – within 0.6–10 %. The highest content of impurities and ED-24 resin (10 %) was found for HPO.

MS-method additionally confirms the results of chemical analysis and IR- and NMR-spectroscopy concerning the structure of some hydroxy derivatives of ED-24 epoxy resin.

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МЕТОДИ ОФ-ВЕРХ ЕС-МС і УФ ДЛЯ ВИЗНАЧЕННЯ ХРОМАТОГРАФІЧНИХ ПІКІВ ДЕЯКИХ ГІДРОКСИПОХІДНИХ ЕПОКСИДНИХ СМОЛ

Анотація. *З використанням методу обернено-фазової високоефективної рідинної хроматографії з мас-спектрометричним детектуванням (ОФ-ВЕРХ ЕС-МС) вивчено склад продуктів, синтезованих на основі епоксидної смоли ЕД-24, що містять у своїй структурі вільні гідроксильні групи та інші, зокрема епоксидну, пероксидну, карбоксильну чи акрилатну групи.*

Ключові слова: *епоксидна смола, олігомери з функційними групами, ОФ-ВЕРХ, ЕС-МС.*