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## PREPARATION OF 9-HYDROXYNONANOIC ACID METHYL ESTER BY OZONOLYSIS OF VEGETABLE OILS AND ITS POLYCONDENSATION

*Vegetable oil-based and potentially biodegradable polyesters were prepared from 9-hydroxynonanoic acid methyl ester. This paper describes ozonolysis of vegetable oils and the method for preparation of useful monomers and in particular 9-hydroxynonanoic acid methyl ester. Ozonolysis of soybean oil and castor oil in methanol and methylene chloride solution, followed by reduction with sodium borohydride was used to obtain a mixture of triols, diols and monols. Triglyceride triols were separated from the rest of the mixture and transesterified with methanol to obtain methyl esters of fatty acids and glycerin. The main component of fatty acids was 9-hydroxynonanoic acid methyl ester, which was characterized and used for polycondensation by transesterification. High molecular weight polyhydroxy alcanoate was a solid having a melting point of 75 °C. The molecular weight of the resulting polyester was affected by the purity of the monomer and side reactions such as cyclization. The polymer was characterized by chromatographic, thermal and analytical methods.*

New technologies which consider use of renewable, biobased and more cost-effective resources in the production of polymeric materials gave some valuable results. Vegetable oils were found to be a good replacement for petroleum for the preparation of chemicals and a number of monomers from vegetable oils have been already reported [1–7].

Vegetable oils are triglycerides of fatty acids. There are several techniques which can be used to functionalize unsaturated vegetable oils in order to provide starting materials for further polymerization to polyurethane elastomers [8–11].

Concerning terminology we will call ozonation the process of treating a material with ozone while ozonolysis is the cleavage of an alkene to carbonyl compounds.

Ozonation followed by reduction is a very efficient technique for carbon–carbon double bond cleavage in unsaturated oils that can give polyols or monomers of well-defined structure [9]. Utilization of ozonolysis for obtaining such products has been studied for a long time [12].

Ozonolysis followed by reduction of the soybean and castor oils was used in our study. Polyols with terminal primary hydroxyl groups, useful as valuable intermediates for the chemical and plastics industries were obtained.

Methanolysis of the ester bonds of the obtained polyols, followed by purification can be used for obtaining 9-hydroxynonanoic acid methyl ester. A method for the production of hydroxycarboxylic acids involving coupling a fatty acyl group by enamine chemistry followed by a ring expansion and selective reduction of keto acids

has been given in a European patent [13]. This monomer is highly reactive due to the presence of the primary hydroxyl group and can have numerous applications [14]. In this paper, we present the study for preparation of 9-hydroxynonanoic acid methyl ester from soybean and castor oils and its polycondensation by transesterification to obtain high molecular weight polyhydroxy alcanoate. Fatty acid profiles for the soybean and castor oils are given in Table 1.

Table 1. Composition (%) of the major fatty acids in the soybean and castor oil

Tabela 1. Sastav (%) osnovnih masnih kiselina u sojinom i ricinusovom ulju

Acid	Soybean oil	Castor oil
C16:0	10.9	1.0
C18:0	4.2	1.0
C18:1	24.9	3.0
C18:2	52.7	4.0
C18:3	6.2	0.3
C18:1+OH	-	89.5
C18:1+2OH	-	0.7

Typical soybean oil has two dominating fatty acids with 18 carbon atoms – linoleic acid with two double bonds and oleic acid with one double bond. There is also about 3–10% of linolenic acid with three double bonds, along with about 15% saturated acids.

The total ozonolysis of an oil molecule followed by the reduction will produce a triglyceride consisting of the C9 fatty acids with terminal hydroxyl groups (Figure 1).

The saturated fatty acids are left unaffected by ozonolysis and their relative weight content increases to about 22.5% in the ozo-SOY-polyol. Their presence decreases the functionality and hydroxyl number of the ozo-SOY-polyol while increasing molecular weight. The byproducts produced include: 1-nonanol, 1-hexanol, 1-propanol and 1,3-propane diol.

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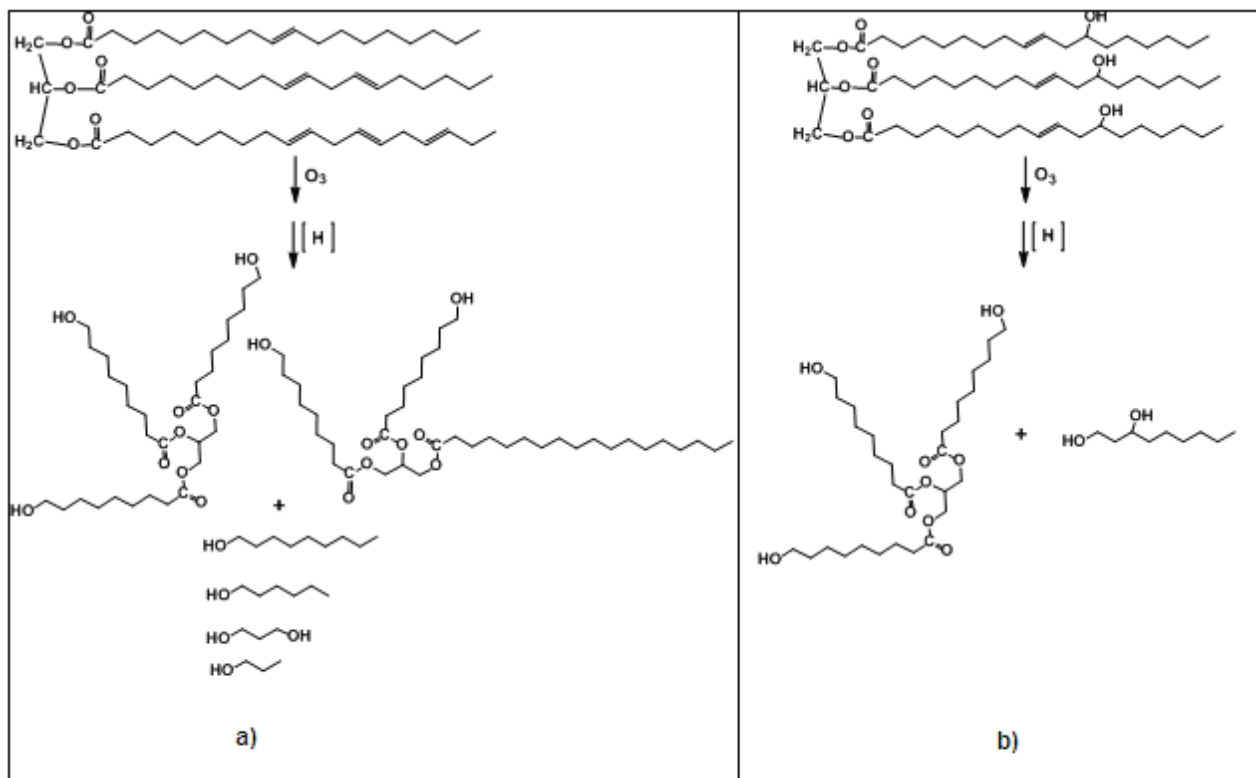


Figure 1. Products of vegetable oil ozonolysis: (a) soybean oil; (b) castor oil.  
Slika 1. Proizvodi ozonolize biljnih ulja: (a) sojino ulje; (b) ricinusovo ulje.

Castor oil, on the other hand, has one dominant fatty acid – ricinoleic acid, and very low content of the saturated acids (2%). In the ozo-CO-polyol from castor oil, relative weight content of the saturates increases up to 3.16%. Byproducts include mainly 1,3-nonanediol and a very small quantity of low molecular weight compounds.

Ozonolysis of the soybean and castor oil has been carried out under the same conditions in the methylene chloride/methanol solution. Methylene chloride is an inert solvent, while the presence of methanol is important since it reacts with the intermediate dipolar carbonyl oxide (Figure 2) to give hydroperoxide. The hydroperoxide is much more easily reduced to carbonyl compounds than the cyclic peroxide (Staudinger ozonide), which is formed if methanol is not present. Therefore, methanol is considered as a reactive solvent in the ozonolysis process [12]. Reaction products are hydroperoxides and aldehydes which are reduced to the hydroxyl groups in the next reaction step.

Ozonolysis can be defined as a breakdown of the ozonation products. Oxidative cleavage of the ozonation products gives acids or ketones, while reductive cleavage gives primary and secondary alcohols or aldehydes and ketones [12].

Reduction of ozonolysis products has been carried out with sodium borohydride. Reduction can be also

carried out using zinc/acetic acid solution, triphenylphosphine, dimethyl sulphide or catalytic hydrogenation in the presence of Raney nickel catalyst [14]. It was also found recently that electrochemical reduction [6] can significantly reduce reduction costs.

Sodium borohydride is probably one of the most useful reducing agents available today. The presence of the protic solvents (*i.e.* 2-propanol and methanol) modifies its reducing characteristics by introducing alkoxy groups onto  $\text{NaBH}_4$  with the formation of  $\text{Na}^+[\text{B}(\text{OR})_x\text{H}_y]^-$ , where  $x + y = 4$ , involving the solvent in the transfer of the hydride ion to the carbonyl group [15,16]. Alkoxy groups on  $\text{NaBH}_4$  increase the electron density on the central boron atom through resonance effects, thus making it a better reducing agent.

Figure 3 illustrates classical organic reductions accomplished in protic solvents. In these reductions,  $\text{NaBH}_4$  attacks the carbon atom with the largest positive charge [17].

Wigfield has summarized the mechanism of hydride reduction in an interesting article [18], saying that the most probable mechanism for ketone reduction is the acyclic mechanism where the borohydride anion delivers a hydride ion to the carbonyl carbon at the same time that a proton is transferred from the solvent to the carbonyl oxygen (Figure 4).

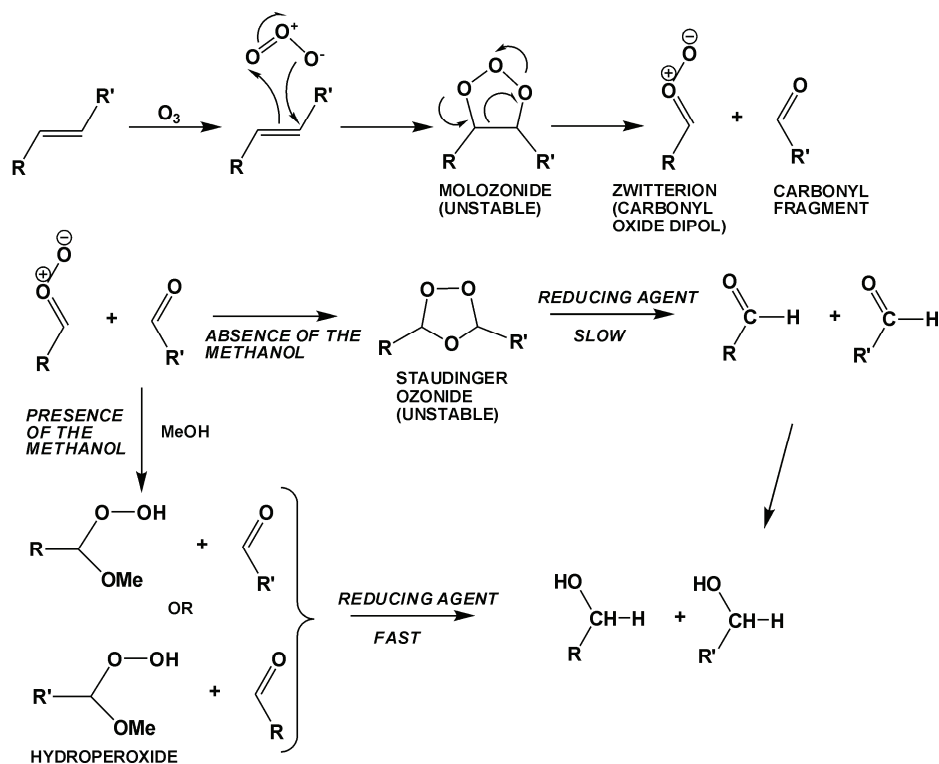


Figure 2. Ozonation and reduction mechanism.

Slika 2. Mehanizmi ozonizacije i redukcije.

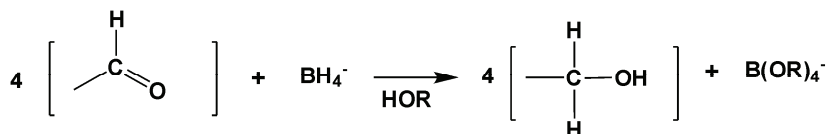


Figure 3. Reduction of the carbonyl groups in the presence of sodium borohydride and protic solvent.

Slika 3. Redukcija karbonilnih grupa u prisustvu natrijum borohidrida i rastvarača-donora vodonikovih jona.

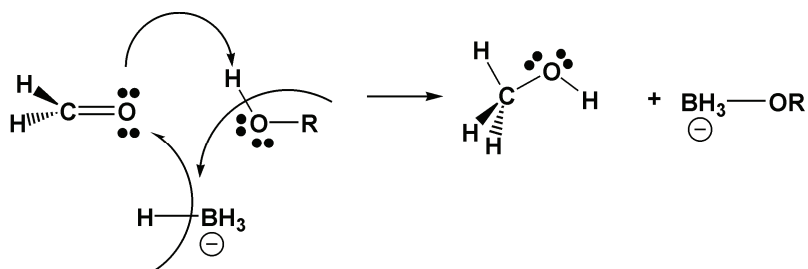


Figure 4. Mechanism of reduction of carbonyl groups in the presence of the borohydride and alcohol solvent.

Slika 4. Mehanizam redukcije karbonilnih grupa u prisustvu borohidrida i alkoholnog rastvarača.

Four moles of a carbonyl compound are reduced directly to alcohol by one molecule of  $\text{NaBH}_4$ , with the protic solvent participating in the reduction [16–19].

After completion of the reaction, products are washed with water to remove reducing agent decomposition products (borate, sodium hydroxide, boric acid), as well as low molecular weight side products. Still, higher molecular weight impurities (*n*-nonanol or 1,3-nonanediol) have to be removed by distillation in order to obtain pure ozo-SOY-polyol and ozo-CO-polyol.

## EXPERIMENTAL SECTION

### Materials

Soybean oil having an iodine value (IV) 129 was supplied by Cargill, Minneapolis, MN. Castor oil having iodine value 83 and hydroxyl number 168 mg KOH/g was purchased from Alfa Aesar. Methanol (HPLC), methylene chloride (HPLC), sodium sulfate (anhydrous), and ethyl ether (laboratory grade) were purchased from Fisher Scientific, Pittsburgh, PA. Sodium borohydride

(98%) was obtained from Aldrich, Milwaukee, WI. Titanium (IV) isopropoxyde, >98%, was purchased from Acros Organics.

### Methods

The IR spectra of the ozo-SOY-polyols were recorded on a Perkin Elmer Spectrum-1000 Fourier transform infrared (FTIR) spectrometer.

The GPC chromatograms were acquired on a Waters system consisting of a 510 pump and 410 differential refractometer. Tetrahydrofuran was used as eluent at a flow rate of 1.00 ml/min at 30 °C. Four Phenogel columns plus a guard Phenogel column from Phenomenex covering a MW range of  $10^2$  to  $5 \times 10^5$  were used.

The hydroxyl values of the polyols were determined according to the ASTM E 1899-97 standard test method for hydroxyl groups using reaction with p-toluenesulfonyl isocyanate (TSI) and potentiometric titration with tetrabutylammonium hydroxide.

Acid values were measured using standard test method for fats and oils.

Differential scanning calorimeter, model Q100, from TA Instruments, New Castle, DE, USA, was used to measure melting at a heating rate of 20 °C/min.

### Synthesis of polyols *via* ozonolysis

Ozonolysis of the soybean and castor oil was carried out by introducing ozone/oxygen at the flow rate of 1 l/min. Solution of 10% soybean oil in a mixture of methanol and methylene chloride (volume ratio 45/55) is used. The ozonation apparatus is presented on Figure 5. Ozone is generated from the molecular oxygen by electric discharge in the ozone generator (Clear Water, Tech INC. M-1500). The ozone concentration (wt%) in the oxygen stream was monitored using an ozone analyzer (Mini Hicon, SC-010-R, IN USA, Inc.). Maximum ozone concentrations in the oxygen stream was 5.8 wt%.

The reaction mixture is maintained at 0–3 °C, using an ice/water bath. Higher reaction temperatures favor formation of undesired oligomeric products. Ozonolysis is finished after the concentration of ozone monitored by the ozone analyzer becomes significant. At the same time in the dry ice/acetone trap, that contains some methanol removed from reaction mixture, royal blue color appears. Longer reaction time can cause further oxidation of the formed aldehydes to carboxylic acids. Ozonolysis products in this case would have lower hydroxyl number and higher acid value.

Prior to the reduction, the reaction mixture was purged with nitrogen for 10 min to remove peroxides.

Reduction has been carried out at reaction temperature (0–3 °C) by slowly adding sodium borohydride (molar ratio 1.05/1 of reducing agent per double bond equivalent). After adding hydride, stirring was continued for an additional 10 min. Shorter reduction time is sufficient for completing the reaction, while longer reduction times may cause splitting of ester bonds in triglycerides due to the basic environment.

Polyol solutions were washed with water until the water phase was no longer basic. Using brine solution in the first wash enhanced separation, which is slow and difficult due to the presence of a large concentration of hydrophilic hydroxyl groups. After washing, the organic phase was dried over anhydrous sodium sulfate. Solvents and low molecular weight products were removed under reduced pressure at 80 °C. Obtained polyols still contain impurities that could not be removed during synthesis (1-nonanol and 1,3-nonanediol).

### Purification of the ozo-polyols

Ozo-polyols were purified using a thin-film evaporator (Wiped Film-Still, POPE Scientific, Inc.). Separation has been carried out at 120 °C and 0 mm Hg.



Figure 5. Ozonation apparatus.  
Slika 5. Aparatura za ozonizaciju.

### Preparation of 9-hydroxynonanoic acid methyl ester

Ozo-CO-polyol has been used for the preparation of 9-hydroxynonanoic acid methyl ester by methanolysis. Reaction was catalyzed by 1% CH<sub>3</sub>OK. A large excess of methanol (15:1 molar ratio of methanol per double bond of triglyceride) was used in the process. The reaction was carried out under reflux of methanol, at 68 °C for 3 h. After cooling to room temperature, diethyl ether was added to the mixture, and then washed with water several times, until the pH value of the water phase was 7. The organic phase was dried using anhydrous sodium sulfate, and ether was removed on a rotary evaporator. Pure 9-hydroxynonanoic acid was obtained from the mixture as a distillate, using short path high vacuum distillation, where the residue after distillation contains mainly methyl esters of the saturated fatty acids.

### Polymerization of 9-hydroxynonanoic acid methyl ester by transesterification

Polyhydroxy alcanoate has been prepared using polycondensation of 9-hydroxynonanoic acid methyl ester in the presence of 0.5 wt% titanium (IV) isopropoxyde catalyst. Reaction took place in a round bottom flask at 150 °C, with mechanical stirring and N<sub>2</sub> bubbling. Methanol was removed within the first half hour and the reaction was continued for 4 h until viscosity was too high to permit good mixing.

## RESULTS AND DISCUSSION

### Properties of the Ozo-polyols

Gel permeation chromatogram of the soybean oil ozonolysis products is shown in Figure 6. The polyol

has two main peaks, where the right one is assigned to the triglyceride of 9-hydroxynonanoic acid, and the left one presents the triglycerides containing saturated fatty acids together with 9-hydroxynonanoic acid methyl ester (structure a in Figure 1). The small peak with a retention time of about 36.5 min is 1-nonanol that could not be removed after synthesis.

Ozo-SOY-polyol and ozo-CO-polyol after removing 1,3-nonanediol and/or 1-nonanol are presented in Figure 7. The polyol obtained from castor oil has also the main peak assigned to the triglyceride of 9-hydroxynonanoic acid. The peak on the left side is much smaller compared to the ozo-SOY-polyol due to lower concentration of saturated fatty acids in castor oil. The peak on the right side results from diglycerides obtained by the hydrolysis of triglycerides after washing the reaction mixture in the basic environment. Formation of the diglycerides is related to the washing step, where hydrolysis of the ester bonds may occur in basic environment. The washing step was significantly longer in the case of ozo-CO-polyol, due to very slow separation of the organic and water phases. Probably the presence of the larger concentration of the hydrophilic hydroxyl groups in ozo-CO-polyol, compared to ozo-SOY-polyol influenced slower separation, longer washing time and, as a consequence, a larger possibility of diglyceride formation. Theoretical and measured hydroxyl number values, acid number values, as well as theoretical number average molecular weight ( $M_n$ ), and ozo-polyol functionalities are summarized in Table 2. The FTIR spectra of the ozo-polyols are presented on the Figure 8.

The synthesized ozo-polyols have hydroxyl numbers very close to the theoretical values. Before remov-

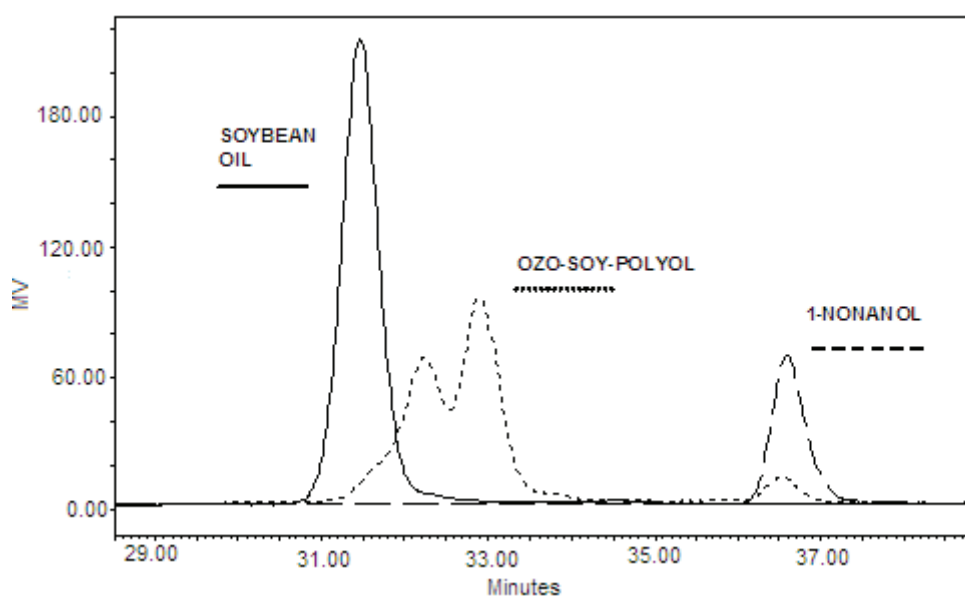


Figure 6. GPC chromatograms of soybean oil and the polyol obtained by ozonolysis of soybean oil.  
Slika 6. GPC hromatogram sojinog ulja i poliola dobijenog ozonolizom sojinog ulja.

ing impurities using the thin film evaporator, hydroxyl values were significantly higher – 250 mg KOH/g for ozo-SOY-polyol and 450 mg KOH/g for ozo-CO-polyol. Thin layer evaporation is an efficient technique for se-

paration. It provides very fast separation of the components without exposing samples to high temperatures for a long time and therefore prevents loss of the hydroxyl groups due to the dehydration.

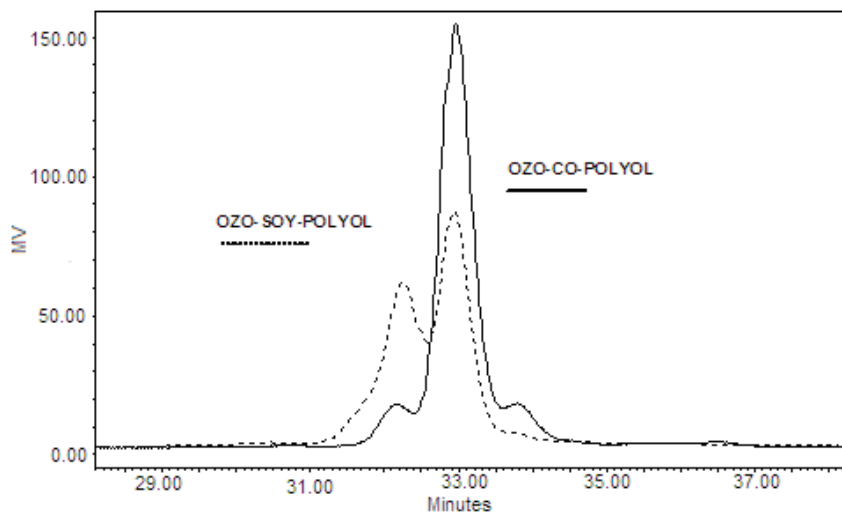


Figure 7. GPC chromatograms of the ozo-SOY-polyol and ozo-CO-polyol.

Slika 7. GPC hromatogram ozo-SOY-poliola i ozo-CO-poliola.

Table 2. General properties of the polyols

Tabela 2. Svojstva poliola

Polyol	Theor. OH #, mg KOH/g	Exp.OH #, mg KOH/g	Acid #, mg KOH/g	Theor. Mn	Theor. functionality, <i>f</i>
Ozo-SOY-polyol	234	234	0.8	603	2.52
Ozo-CO-polyol	287	296	1.4	570	2.92

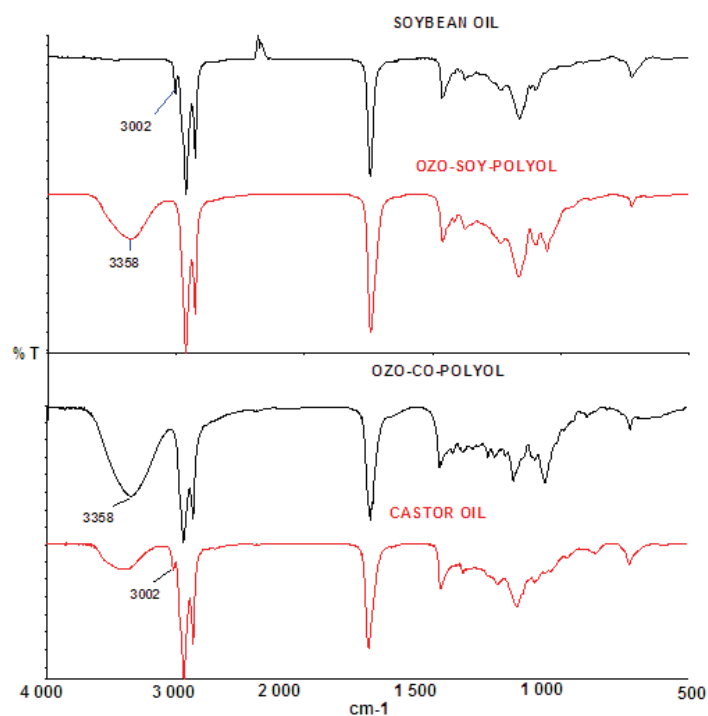


Figure 8. FTIR spectra of the ozo-SOY-polyol and ozo-CO-polyol.

Slika 8. FTIR spektar ozo-SOY-poliola i ozo-CO-poliola.

Relatively low acid values of the polyols indicate that further oxidation of the formed hydroxyl groups did not take place and that the ozonolysis step was successful.

FTIR spectra of the ozo-polyols are very similar. Disappearance of double bonds ( $3002\text{ cm}^{-1}$ ) indicating the completion of the reaction, and appearance/increase

of the hydroxyl group peak ( $3358\text{ cm}^{-1}$ ) is evident in both spectra. A significantly higher hydroxyl peak in the ozo-CO-polyol is evident.

Both polyols are white, partly solid at room (Figure 9). Multiple melting peaks are common for the triglyceride systems.

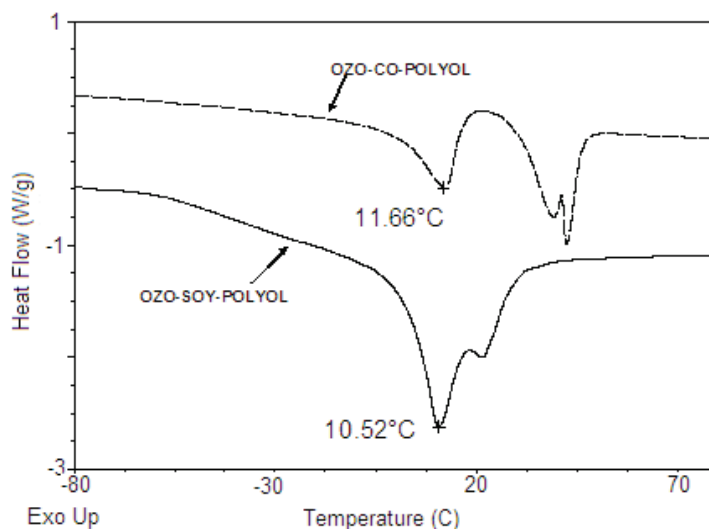


Figure 9. DSC traces of the ozo-SOY-polyol and ozo-CO-polyol.  
Slika 9. DSC krive ozo-SOY-poliola i ozo-CO-poliola.

#### Preparation of 9-hydroxynonanoic acid methyl ester

Methyl ester of 9-hydroxynonanoic acid was obtained from the ozo-CO-polyol by transesterification with methanol. Methanolysis of the ozo-CO-polyol resulted in the slight loss of hydroxyl groups due to the dimerization (Figure 10). Obtained methyl esters of the ozo-CO-polyol had hydroxyl number 274 mg KOH/g,

and acid value 0.5 mg KOH/g. 9-Hydroxynonanoic acid methyl ester was separated from the rest of the mixture (methyl esters of saturated fatty acids) using short path high vacuum distillation. The obtained pure material had hydroxyl number 293 mg KOH/g (theoretical value is 298). The methyl ester of the 9-hydroxynonanoic acid is a clear, colorless, low viscosity liquid. DSC traces are presented in Figure 11.

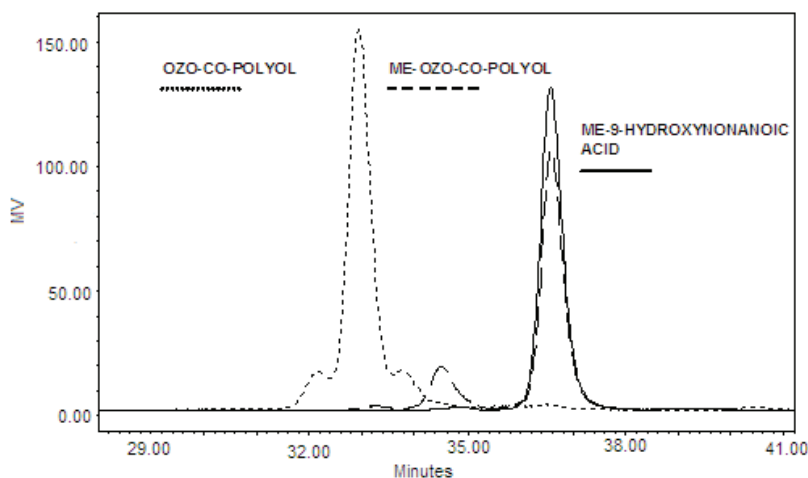


Figure 10. GPC chromatograms of the 9-hydroxynonanoic acid methyl ester, polyol obtained after ozonolysis of castor oil and methyl esters from the polyol.

Slika 10. GPC hromatogram metil estera 9-hidroksinonanske kiseline, poliola dobijenog ozonolizom ricinusovog ulja i metil estara dobijenih od poliola.

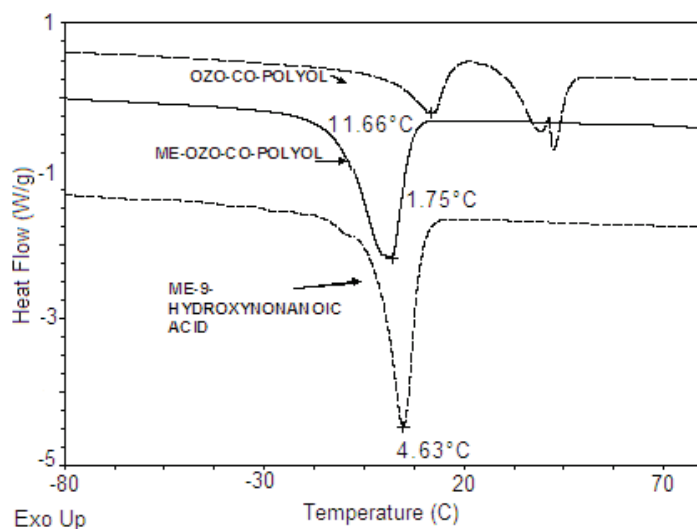


Figure 11. DSC traces of the ozo-CO-polyol, methyl esters from ozo-CO-polyol and pure methyl ester of 9-hydroxynonanoic acid.  
Slika 11. DSC krive ozo-CO-poliola, metil estara dobijenih od ozo-CO-poliola i prečišćenog metil estara 9-hidroksinonanske kiseline.

### Transesterification of 9-hydroxynonanoic acid methyl ester

High molecular weight linear monol was obtained after transesterification of the 9-hydroxynonanoic acid methyl ester. Resulting hydroxyl number indicates 98% conversion of hydroxyl groups. As Table 3 indicates, after 0.5 h of reacting, hydroxyl conversion was already

82%, due to the high reactivity of primary hydroxyl groups in the monomer. Molecular weight calculated based on hydroxyl number is 11680 and broad molecular weight distribution can be observed on the GPC chromatogram (Figure 12). Obtained polyhydroxy alcanoate is white, solid material with melting temperature of 75 °C (Figure 13).

Table 3. Hydroxyl group conversion during transesterification of 9-hydroxynonanoic acid methyl ester  
Tabela 3. Konverzija hidroksilnih grupa tokom transesterifikacije metil estara 9-hidroksinanolne kiseline

Reaction time, h	0.5	1	2	4
OH#, mg KOH/g	52.8	30.1	14.4	4.8
OH# conversion, %	82	90	95	98

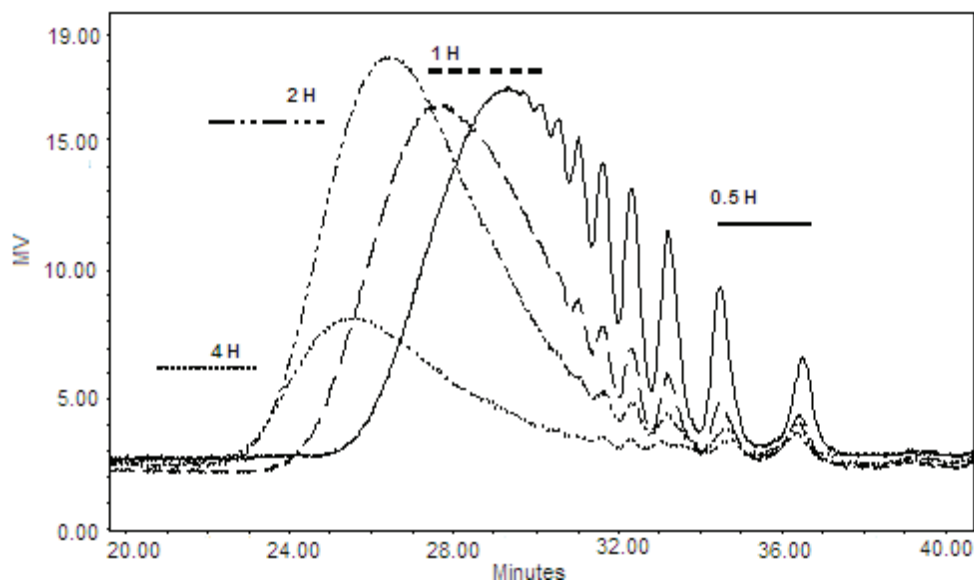


Figure 12. GPC chromatograms of the transesterification products.  
Slika 12. GPC hromatogram proizvoda transesterifikacije.



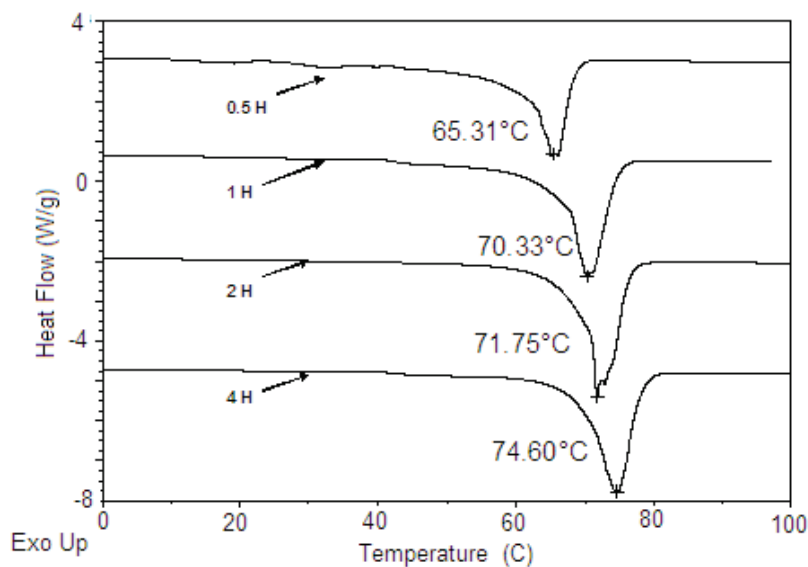


Figure 13. DSC traces off the transesterification products.  
Slika 13. DSC krive proizvoda transesterifikacije.

## CONCLUSIONS

Ozonation followed by reductive ozonolysis has been successfully carried out using soybean and castor oil as starting materials. Obtained ozo-polyols have properties as predicted. Ozo-CO-polyol has higher functionality due to the lower content of the saturates, and it is an excellent source for the 9-hydroxynonanoic acid methyl ester, an interesting monomer for polyesters synthesis. The methyl ester of 9-hydroxynonanoic acid was obtained with good purity and it was utilized in the synthesis of the high molecular weight polyhydroxy alcanoate by transesterification.

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**IZVOD****DOBIJANJE METIL ESTRA 9-HIDROKSINONANONSKE KISELINE OZONOLIZOM BILJNIH ULJA I NJEGOVA POLIKONDENZACIJA**

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Naučni rad

Potencijalno bio-razgradljivi poliestri na bazi biljnih ulja su dobijeni od metil estra 9-hidroksinonanonske kiseline. U ovom radu su detaljno opisani postupak ozonolize biljnih ulja kao i metod za dobijanje korisnih monomera sa posebnim akcentom na dobijanje metil estra 9-hidroksinonanonske kiseline. Sojino i ricinusovo ulje su ozonolizovani u rastvoru metanola i metilen hlorida, a zatim je reakcioni proizvod redukovan borhidridom da bi se dobila smeša triola, diola i mono-hidroksilnih alkohola. Triglicerid trioli su razdvojeni od ostatka smeše i izvršena je transesterifikacija sa metanolom gde su krajnji proizvodi estri masnih kiselina i glicerina. Kao glavna komponenta masne kiseline dobijen je metil estar 9-hidroksinonanonske kiseline koji je dalje ispitan i potom korišćen u polikondenzaciji putem transesterifikacije. Dobijeni polihidroksi alkanoat velike molekulske mase je čvrsta supstanca sa temperaturom topljenja 75 °C. Na molekulsku masu poliestra uticali su čistoća monomera i paralelne reakcije kao što je ciklizacija. Za karakterizaciju polimera korišćene su hromatografske, termičke i analitičke metode.

Ključne reči: Ozonoliza • Metil estar 9-hidroksinonanonske kiseline • Obnovljive sirovine • Poliestar

Key words: Ozonolysis • 9-Hydroxynonanoic acid methyl ester • Renewable resources • Polyester