

Rapeseed Oil as Renewable Resource for Polyol Synthesis

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Abstract – Vegetable oils are one of the most important platform chemicals due to their accessibility, specific structure of oils and low price.

Rapeseed oil (RO) polyols were prepared by amidization of RO with diethanolamine (DEA). To determine the kinetics of amidization reaction, experiments were carried out. Fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), amine (NH) value was determined. Group contribution method by Fedor's was used to calculate solubility parameters, van der Waals volume was calculated by Askadskii. Obtained polyol's OH and NH value are from 304 up to 415 mg KOH/g. RO polyols synthesis meets the criteria of "green chemistry".

In the present study, reaction of RO amidization with DEA was investigated, as well as optimum conditions for polyol synthesis was established to obtain polyols for polyurethane production. Calculations of solubility parameter and cohesion energy density were calculated, as RO polyols will be used as side chains in polymers, and solubility parameter will be used to explain properties of polymers.

Keywords – amidization, esterification, NH value determination, polyols, rapeseed oil, renewable resources, and vegetable oil

I. INTRODUCTION

Petrochemical resources, used intensively in the chemical industry worldwide, are limited and are rapidly decreasing. The chemical industry is making big efforts to find alternatives for petrochemical raw materials, and environmental pressures demand cleaner processes which result in active development of "green" chemistry – using cleaner processes, less energy and renewable materials [1, 2, 3].

Increasing attention is paid to obtaining polymer materials from renewable resources. Natural oil-derived polyols have

been considered as an alternative for petroleum-based polyols. Extended usage of different vegetable oils for various applications is important not only because of diminishing petroleum resources, an important aspect is also environmental protection and sustainability.

Vegetable oils are triglycerides of fatty acids, which have a number of excellent properties, so they could be utilized in producing valuable polymeric materials and, probably, will become a potential bio-renewable feedstock for polyurethanes [4, 5, 6, 7]. Vegetable oils consist of triglycerides which are made up of three fatty acids joined to a glycerol backbone (Figure 1). Most of the common oils contain fatty acids that vary from 14 to 22 carbons in length, with 0 to 3 double bonds per fatty acid, popular fatty acids are with chain lengths of C16 - C18 [1, 5, 8].

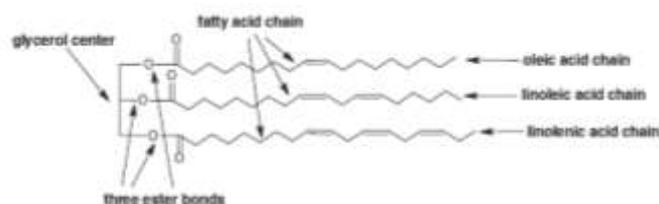


Fig. 1. The triglyceride chain containing three fatty acid chains joined by a glycerol center [5].

Polyols play an important role in polyurethane industry. To obtain polyurethane materials from vegetable oil, hydroxyl groups are necessary for the reactions with isocyanate groups that result in urethane linkages. To obtain polyurethane materials from vegetable oil, hydroxyl groups are necessary for the reactions with isocyanate groups that result in urethane linkages. Unfortunately, there are no hydroxyl groups in RO triacylglycerol structures of saturated and unsaturated fatty

TABLE I
FATTY ACID COMPOSITION (%) OF VARIOUS PLANT OILS [ADAPTED FROM 4]

Carbon atoms: Double bonds	14:0	16:0	16:1	18:0	18:1	18:2	18:3	20:0	20:1	22:0	22:1	24:0
Rapeseed oil	0.1	4.0	0.3	1.8	60.9	21.0	8.8	0.7	1.0	0.3	0.7	0.2
Soybean oil	0.1	10.6	0.1	4.0	23.3	53.7	7.6	0.30	0	0.3	0	0
Linseed oil	0	6.0	0	4.0	22.0	16.0	52.0	0.5	0	0	0	0
Castor oil	0	2.0	0	1.0	7.0	3.0	0	0	0	0	0	0
Palm oil	1.0	44.4	0.2	4.1	39.3	10.0	0.4	0.3	0	0.1	0	0
Sunflower oil	0.1	7.0	0.1	4.5	18.7	67.5	0.8	0.4	0.1	0.7	0	0
Palm kernel oil	16.2	8.4	0	2.5	15.3	2.3	0	0.1	0.1	0	0	0
Corn oil	0.1	10.9	0.2	2.0	25.4	59.6	1.2	0.4	0	0.1	0	0

acids; hence, there is a need for chemically introduced hydroxyl groups onto RO triacylglycerol's [9]. Rapeseed oil or canola oil, as called in North America, is derived from *Brassica napus L.* oilseeds. RO mainly consists of unsaturated fatty acids – oleic, linoleic acid, linolenic acid and other saturated and unsaturated fatty acids (Table I).

The literature presents several chemical pathways to introduce hydroxyl functionality into the triacylglycerol's of vegetable oils containing unsaturated fatty acids [7, 10, 11, 12].

Natural oil polyols with primary hydroxyl groups can be synthesized by vegetable oil reacting with amino alcohols [13].

The focus of this research is to determine kinetics of RO amidization with DEA and to understand what changes happen to polyol when it is stored for a longer period of time. Calculation on solubility parameters (δ) of vegetable oils and fatty acids and cohesion energy density (e_{coh}), as well as van der Waals volume (V_w) of fatty acid, were carried out.

II. EXPERIMENTAL PROCEDURES

A. Materials

Rapeseed oil (specifications: iodine value = 117 I₂ mg/100 g sample, acid value = 2.1 mg KOH/g, saponification value = 192 mg KOH/g sample) was obtained from Iecavnieks, Latvia. The fatty acid composition was as follows: 62.4 % oleic acid, 20.6 % linoleic acid, 9.3 % linolenic acid, 4.7 % palmitic acid and 0.3 % other fatty acids. Diethanolamine 99.2 % (Huntsman, The Netherlands) was used as purchased.

B. Synthesis of rapeseed oil polyols

Polyols of RO were prepared by amidization. The reaction was carried out in a three neck 1.0 L thermo resistant glass reaction flask submerged in a silicone bath. The reaction flask was equipped with a mechanical stirrer, a thermometer, a cooler and an argon inlet tube. RO (1 M) was weighed and heated to 140 ± 5 °C, then 0.15 wt % zinc acetate as catalyst was added with stirring and during the next 15 min 139 g (2.1 M) of preheated (to 70 °C) DEA was added. The reaction of amidization was carried out for 4 - 5 h. The end of reaction is tested by solubility of obtained product in ethanol at volume ratio 1:1. If the RO polyol dissolves in ethanol, it is the end of the amidization. If needed, vacuum distillation at 200 mmHg is used to remove water from synthesis, for example, if polyol is used for obtaining polyurethane films. RO diethanolamides at different molar ratios were prepared as described.

C. NH value determination

NH value was determined by titration of the sample (0.3 - 0.4 g RO polyol) in ethanol solution with standardized 0.1 N HCl by using bromoresol green solution as an indicator.

D. FTIR Spectra Analysis

FTIR spectra measurements were made on a Perkin-Elmer spectrometer, Model: Spectrum One FTIR Spectrometer. FTIR spectroscopy data were used to monitor functional groups of the synthesized products.

E. Differential scanning calorimetry

Calorimetric measurements (DSC) were made on a differential scanning calorimeter Mettler Toledo 823°. About 3 mg of sample were used to analyse the course of reaction. Experiments were carried out isothermally at 120 °C, 140 °C and 160 °C for up to 180 min. The results were plotted as a conversion degree against time in minutes.

III. RESULTS AND DISCUSSION

To characterize RO solubility parameter, cohesion energy density and van der Waals volume were calculated. Solubility parameter and cohesion energy density were calculated by Fedor's group contribution method [14], van der Waals volume by Askadskii [15, 16]. The results are shown in Table II.

TABLE II
SOLUBILITY PARAMETERS, COHESION ENERGY DENSITY, VAN DER WAALS VOLUME OF SOME VEGETABLE OILS

Vegetable oil	Solubility parameter, (cal/cm ³) ^{1/2}	Cohesion energy density, (cal/cm ³) ^{1/2}	van der Waals volume, cm ³ /mol
Rapeseed oil	8.94	80.00	184.02
Soybean oil	8.96	80.28	177.90
Linseed oil	8.99	80.83	176.54
Castor oil	10.03	100.52	187.23

For soybean oil, the calculated solubility parameter 8.94 (cal/cm³)^{1/2} is close to (δ) calculated by King by inverse gas chromatography [17].

Castor oil has slightly higher solubility parameter and cohesion energy density because ricinoleic acid contains OH group which has a much higher energy of vaporization which results in higher cohesion energy density and solubility parameter, respectively.

Also solubility parameter for DEA was calculated. It is 13.45 (cal/cm³)^{1/2}. There is a noticeable difference between solubility parameters of RO and DEA, because of that, RO amidization reaction initially occurs at heterogeneous phase.

The composition of vegetable oil and fatty acid content differs within few per cent, depending on weather and other factors. Solubility parameters of unsaturated and saturated fatty acid radicals, attached to glycerol backbone, were calculated (Table III).

As seen in Table III, with the increasing number of CH₂ groups in a fatty acid molecule, the solubility parameter decreases. The solubility parameter of fatty acid radicals plays an important role because synthesized polyols will be used as side chains in polymers. The value of the solubility parameter will affect the effectiveness of solubility, provide better reaction with foaming agents, like pentane ($\delta=7.1$ (cal/cm³)^{1/2}) and freon ($\delta=7.25$ (cal/cm³)^{1/2}), because their solubility parameters are very close [18].

TABLE III
SOLUBILITY PARAMETER OF SATURATED AND UNSATURATED FATTY ACID RADICALS

Name	Formula	Molar mass, g/mol	Carbon atoms: double bond	Structural formula	δ , (cal/cm ³) ^{1/2}
Myristic acid	C ₁₄ H ₂₇ O ₂	227.37	14:0	CH ₃ (CH ₂) ₁₂ COO	8.95
Palmitic acid	C ₁₆ H ₃₁ O ₂	255.42	16:0	CH ₃ (CH ₂) ₁₄ COO	8.90
Palmitoleic acid	C ₁₆ H ₂₉ O ₂	253.41	16:1	CH ₃ (CH ₂) ₅ CH=CH(CH ₂) ₇ COO	8.93
Stearic acid	C ₁₈ H ₃₅ O ₂	283.48	18:0	CH ₃ (CH ₂) ₁₆ COO	8.87
Oleic acid	C ₁₈ H ₃₃ O ₂	281.46	18:1	CH ₃ (CH ₂) ₇ CH=CH(CH ₂) ₇ COO	8.89
Linoleic acid	C ₁₈ H ₃₁ O ₂	279.45	18:2. n-6	CH ₃ (CH ₂) ₄ CH=CHCH ₂ CH=CH(CH ₂) ₇ COO	8.91
Linolenic acid	C ₁₈ H ₂₉ O ₂	277.43	18:3. n-3	CH ₃ CH ₂ CH=CHCH ₂ CH=CHCH ₂ CH=CH(CH ₂) ₇ COO	8.93
Ricinoleic acid	C ₁₈ H ₃₃ O ₃	297.46	18:2	CH ₃ (CH ₂) ₅ CH(OH)CH ₂ CH=CH(CH ₂) ₇ COO	10.18
Arachidic acid	C ₂₀ H ₃₉ O ₂	311.53	20:0	CH ₃ (CH ₂) ₁₈ COO	9.00
Eicosenoic acid	C ₂₀ H ₃₇ O ₂	309.52	20:1	CH ₃ (CH ₂) ₇ CH=CH(CH ₂) ₉ COO	8.86
Behenic acid	C ₂₂ H ₄₄ O ₂	339.59	22:0	CH ₃ (CH ₂) ₂₀ COO	8.84
Erucic acid	C ₂₂ H ₄₁ O ₂	337.57	22:1	CH ₃ (CH ₂) ₇ CH=CH(CH ₂) ₁₁ COO	8.83
Lignoceric acid	C ₂₄ H ₄₇ O ₂	367.64	24:0	CH ₃ (CH ₂) ₂₂ COO	8.82

RO polyols were prepared as described in B. *Synthesis of rapeseed oil polyols*. Obtained RO polyols are characterized by hydroxyl value and NH value (Table IV).

TABLE IV
CHARACTERIZATION OF OBTAINED RAPESEED OIL POLYOLS

Polyol	Molar ratio DEA/RO	Polyols` OH+NH value, (mg KOH/g)
DEA-RO	2.07:1	304
DEA-RO	2.10:1	311
DEA-RO	2.50:1	360
DEA-RO	2.9:1	415

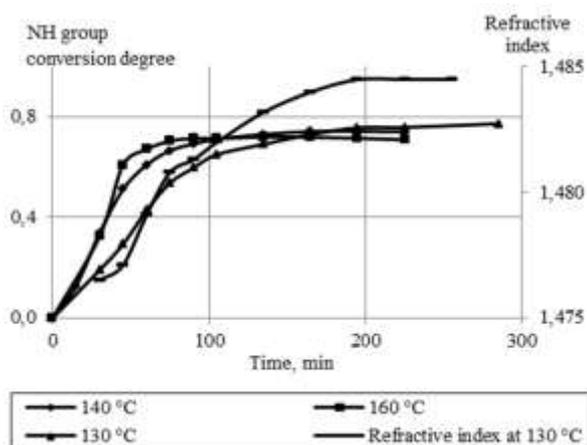


Fig. 2. NH group conversion during RO polyol's synthesis.

To determine kinetics of RO amidization with DEA, RO polyols were synthesized by reacting RO with DEA at a molar ratio of 1:2.9 M. The kinetics of amidization was investigated at three temperatures - 130 °C, 140 °C and 160 °C. The course of reaction was followed and analysed using NH groups' degree of conversion and FTIR spectroscopy data.

Figure 2 shows that during the first hour NH group degree of conversion reaches 0.6, but then the reaction slow down.

At the temperatures of 130 °C, 140 °C and 160 °C, NH groups' conversion degree reached 77 %, 74 % and 72 %, respectively. Also refractive index was controlled during the synthesis and refractive index correlates with increasing NH groups' degree on conversion. Low NH group conversion at the end of the reaction could be explained by thermal rearrangement of the amide to amino ester (Figure 3) [19].

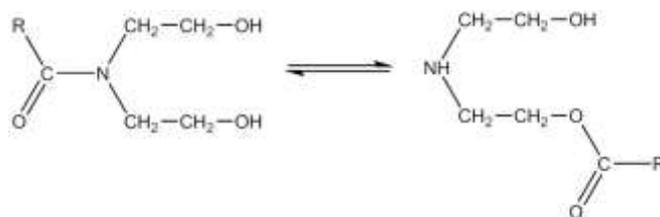


Fig. 3. Reaction products of RO amidization with diethanolamine, where R - C₁₂-C₂₂ are unsaturated and saturated fatty acid radicals.

To test the hypothesis that both amidization and transesterification processes progressed in parallel during the RO diethanolamide synthesis, polyols were stored at room temperature for five to six months and during this period NH value was monitored (Figure 4).

As seen in Figure 4, the NH value of the obtained RO diethanolamides decreases, when they are stored at room temperature for a long time. The greater NH value at the end of the storage period is for the polyols that are synthesized at higher RO and DEA molar ratios.

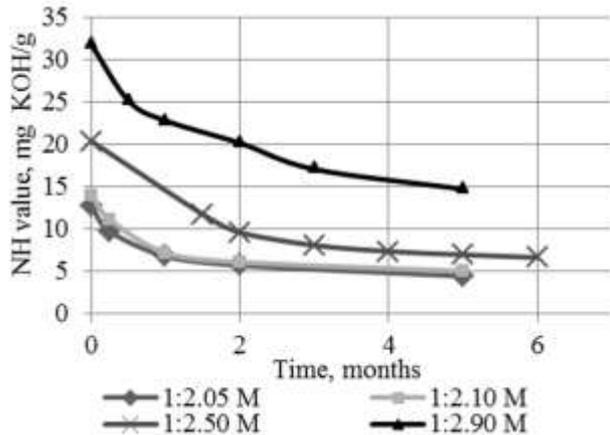


Fig. 4. Changes in NH groups during RO polyol's storage depending on time and RO:DEA molar ratio.

The decreasing of the NH value indicated that there might be equilibrium between diethanolamide and amine group containing compounds. To test the hypothesis whether there is equilibrium between RO diethanolamide and amine group containing compounds, we used RO polyol which was synthesized at RO and DEA molar ratio 1.0:2.5 M and stored for 4 years at room temperature. The polyol was reheated at 140 °C and 160 °C and NH value was monitored (Figure 5)

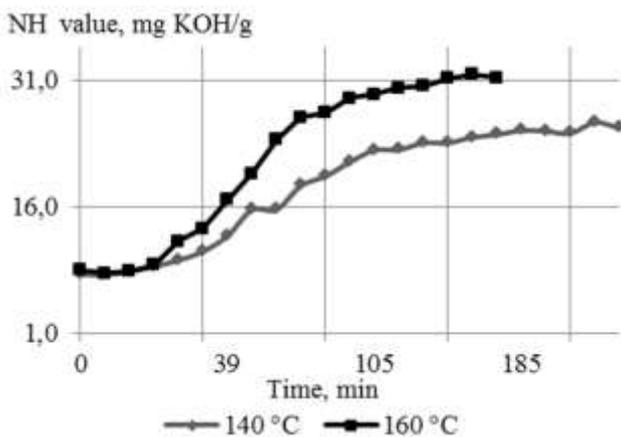


Fig. 5. Change in NH value versus time reheating RO diethanolamide preserved for 4 years, reheated at 140 °C and 160 °C.

As seen in Figure 5, RO polyols NH value was 7 mg KOH/g after storage, after reheating it increased to 27 mg KOH/g at 140 °C, at 160 °C increased to 32 mg KOH/g. The results prove that there is equilibrium between fatty acid amino esters and diethanolamides when RO is amidized with DEA.

The kinetics of RO amidization was investigated also using the Fourier transform infrared spectroscopy data (Figure 6).

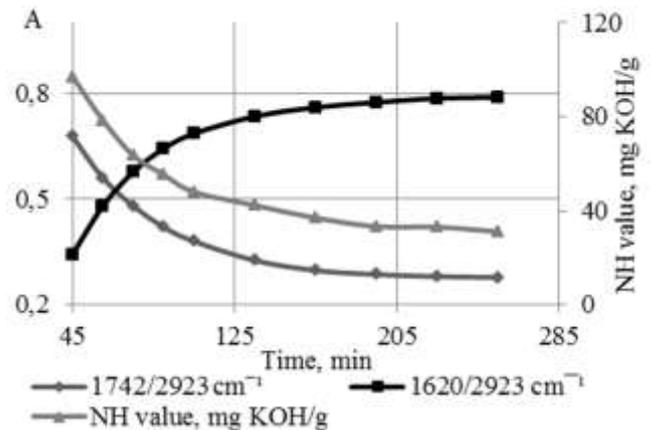


Fig. 6. The ratio of A_{1742}/A_{2923} and A_{1620}/A_{2923} and NH value during diethanolamide synthesis versus time.

The relative intensity of C=O groups decreases at $1739\text{ cm}^{-1}/2923\text{ cm}^{-1}$, while the C=O absorption intensity at $1620\text{ cm}^{-1}/2923\text{ cm}^{-1}$, characteristic of tertiary amide, increases. The results confirm that the NH group degree of conversion reaches 77 % for the synthesis at 130 °C. The NH value correlates with decreasing intensity of C=O groups at $1739\text{ cm}^{-1}/2923\text{ cm}^{-1}$. The NH group conversion degree is expected to be similar also for the synthesis at 140 °C and 160 °C.

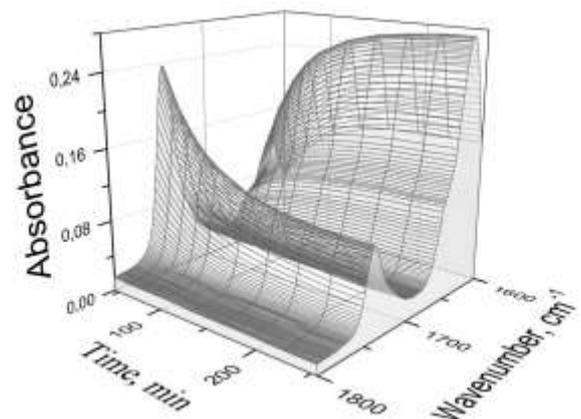


Fig. 7. Time resolved FTIR spectra during polyol's synthesis.

The three dimensional plot shows C=O groups decreasing rapidly during the first 100 minutes and peak at 1620 cm^{-1} , characterizing C=O of tertiary amide, group increasing.

Vegetable oil amidization and transesterification was also analyzed by using differential scanning calorimetry (DSC). Since DEA solubility parameter significantly differs from RO solubility parameter, they form heterogeneous systems. For this reason, DSC experiments were made by using castor oil with DEA at molar ratio 1.0:2.9 M at 140 °C. It was calculated from DSC curves

that the process of castor oil amidization with DEA at 140 °C occurs with the enthalpy 14.53 cal/g.

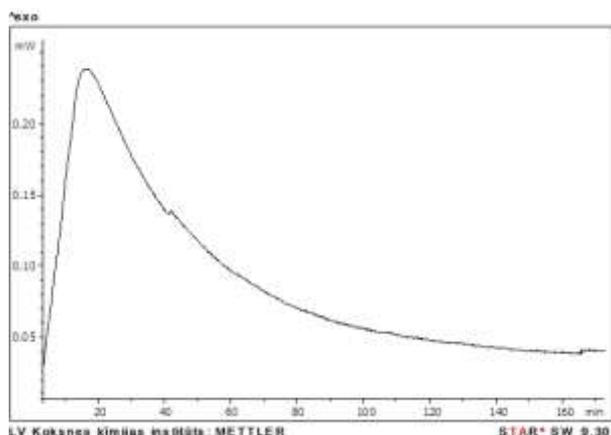


Fig. 8. DSC curve of castor oil amidization with DEA.

IV. CONCLUSIONS

Polyols obtained from RO amidization with DEA at different molar ratios are composed from fatty acid diethanolamide and amino esters. Using FTIR spectroscopy, changes in the concentration kinetics of tertiary amide and ester groups during polyols synthesis process have been established. Equilibrium shifts to fatty acid diethanolamide side, if the temperature of synthesis is lower, and also when polyols are stored. The process of RO polyols synthesis meets the criteria of green chemistry.

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Uldis Stīrna, Anda Fridrihsone, Marija Misāne, Dzintra Vilsone. Rapšu eļļa kā atjaunojama izejviela poliolu sintēzei.

Aktualizējoties vides ilgtspējības jautājumiem un strauji samazinoties fosilo resursu krājumiem, ķīmijas industrija meklē jaunus veidus, kā iegūt izejvielas no atjaunojamiem resursiem. Dažādas augu eļļas tiek uzskatītas par vienu no vissvarīgākajām atjaunojamo izejvielu klasēm, jo tām piemīt pievilcīgas fizikāli – ķīmiskās īpašības, plaši pieejama, salīdzinoši zema cena.

Rapšu eļļas polioli, kas ir izejviela poliuretānu ieguvei, tika iegūti funkcionālizējot rapšu eļļu – ievadot tajā hidroksilgrupas ar amidizācijas reakcijas palīdzību. Polioli tika sintezēti rapšu eļļai reaģējot ar dietanolamīnu dažādās molārās attiecībās no 1:2.05 - 2.9 M pie 140 °C. Iegūtos poliolus raksturo OH un NH skaitlis, kas ir robežās no 304 – 415 mg KOH/g tiek iegūti polioli ar pirmējām hidroksilgrupām. Rapšu eļļas amidizācijas reakcijas kinētika tika pētīta pie molārās attiecības 1:2.9 M pie trīs dažādām temperatūrām 130 °C, 140 °C, 160 °C. Kinētika tika pētīta izmantojot sekojošas metodes - FTIR spektroskopiju, DSC, NH grupu konversiju. NH grupu konversijas pakāpe pie dotajām temperatūrām attiecīgi bija 77 %, 74 % un 72 %. Noskaidrots, ka sintēzes gaitā estera C=O grupas relatīva absorbcijas intensitāte pie A1739/A2923 samazinās, kamēr amīda C=O grupas relatīva absorbcijas intensitāte pie A1620/A2923 palielinās. Noskaidrots, ka paralēli rapšu eļļas amidizācijas procesam, notiek arī tās pāresterifikācijas process, un rodas divi reakcijas produkti: rapšu eļļas amīds un amino grupu saturoša komponente. Poliolu ilgstoši glabājot, NH skaitlis samazinās no 29 līdz 8 mg KOH/g 6 mēnešu laikā, līdzsvars nobīdās uz dietanolamīda pusi. Pēc tam poliolu sildot, NH skaitlis pieaug un līdzsvars nobīdās uz amīna grupu saturošo savienojuma pusi.