A Polyol Composition with Polyurethane End-of-life Material

Field

Embodiments relate to a recycled polyol composition containing polyols that are released from polyurethane End-of-life (EoL) material.

Introduction

A new field in the art of polyurethane technology relates to preparing recycled polyols in which discarded polyurethane (i.e., polyurethane EoL material) is used to create a polyol composition that can be used to make new polyurethane material. Referring to U.S. Publication No. 2023/0399486, it has been proposed that a process for producing a polyol composition containing polyols released from polyurethane EoL material, uses in a reaction mixture in which the reaction mixture includes polyurethane EoL material that is reacted with one or more compounds from the group of (i) polyether polyols having an average molar mass of 200 g/mol to 8000 g/mol and a hydroxyl functionality of 2 to 4, and polyester polyols having an average molar mass of 250 g/mol to 8000 g/mol and a hydroxyl functionality of 2 to 4, (ii) one or more compounds from the group of dicarboxylic anhydrides and dicarboxylic acids, and (iii) water, to form a polyol composition containing polyols released from the polyurethane EoL material. It has been proposed that using such a process allows for the release of polyols from polyurethane being initiated by adding water, without adding a radical former, e.g., a peroxide compound. A unique modification of the process above can be used to specifically recycle an EoL molded high resilience (HR) flexible polyurethane foam (e.g., automotive seating type foam) into a polyol composition especially suitable for the production of molded HR polyurethane foams for automotive applications.

Summary

Embodiments relate to a recycled polyol composition containing polyols released from polyurethane EoL material. The recycled polyol compositions are made by a process which includes providing the polyurethane EoL material and forming a reaction mixture by mixing from 10 wt% to 50 wt%, based on the total weight of the reaction mixture, of polyurethane EoL material. The reaction mixture further includes the following: from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of at least one first polyether polyol comprising an ethylene oxide-capped poly(propylene oxide) polyol having an average hydroxyl number from 20 mg KOH/g to 60 mg KOH/g, from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of one

second polyether polyol comprising either a random copolymer of propylene oxide and/or ethylene oxide or a homopolymer of propylene oxide, and an average hydroxyl number from 40 mg KOH/g to 60 mg KOH/g, from 10 wt% to 15 wt%, based on the total weight of the reaction mixture, of at least one dicarboxylic acid or dicarboxylic acid derivative, from 0.1 wt% to 5 wt%, based on the total weight of the reaction mixture, of at least one short chain polyether diol that is different from the at least one first polyether polyol and the at least one second polyether polyol. The reaction mixture forms the recycled polyol composition, which includes polyols released from the polyurethane EoL material: it has an average hydroxyl number from 30 mg KOH/g to 50 mg KOH/g, and has an average viscosity from 5 Pa·s @ 25 ° C to 15 Pa·s @ 25 ° C.

Detailed Description

The isolation of recycled polyols (also referred to as re-polyols) by the chemical depolymerization of flexible polyurethane foam may be practiced by a range of chemical reaction processes such as glycolysis/alcoholysis, acidolysis, aminolysis, and/or hydrolysis. One particular process utilizes a combination of acidolysis, glycolysis, and hydrolysis to yield a recycled polyol product that contains the isocyanate-derived byproducts (amines, amides, imides, etc.). Preparing a grade of recycled polyol suitable for molded high resilience (HR) polyurethane foam is particularly challenging, as the polyols used to digest the foam must be of a specific equivalent weight per hydroxyl group (EW) that renders lower concentrations of alcohols for foam depolymerization. Such a recipe has not been demonstrated for preparing molded HR recycled polyol compositions. Additionally, prior art does not teach the preparation of a single pot recycled polyol having utility for high resilience molded flexible foam, which is a particular application space that is not tolerant of a broad array of polyols. A single pot recycled polyol is a polyol made in an economically advantageous one-step process where chemical agents (alcohols and glycols, acids, amines, water and the like) break the urethane and/or urea bonds belonging to a flexible polyurethane foam to form a new isocyanate-reactive polyol that may also contain other isocyanate-derived byproducts (amines, amides, imides, etc.).

The embodiments referred to herein have three key components for achieving a molded HR recycled polyol composition (such as a dispersion product) using a combined acidolysis/glycolysis/hydrolysis chemical depolymerization process. First, a high molecular weight ethylene oxide (EO)-capped poly(propylene oxide) polyether polyols led to a recycled polyol dispersion of relatively low OH# with better processability, lower viscosity, a required property in order to produce a good quality molded HR flexible polyurethane foam. Second, the minimum quantity of dicarboxylic acid or dicarboxylic acid derivative digestion agent had to be

significantly increased to isolate a recycled polyol product having a desirable low viscosity for processing. Third, the recycled polyol composition prepared using EoL molded high resilience (HR) flexible polyurethane foam can be used to make a molded HR foam article which exhibits better physical properties than a foam prepared using recycled polyol composition derived from EoL conventional (mattress) flexible polyurethane foam. It is noted that the recycled polyol composition is a dispersion, in which the specific composition has distributed solid particles that are dispersed in a continuous liquid phase.

By polyurethane EoL material we refer here to PU foam that is either post-production recycled materials or post-consumer recycle materials, e.g., in the form of foam trims obtained during molded foam production or in the form of EoL automotive seating PU foam.

By EoL molded HR flexible polyurethane foam we refer here to foam manufactured in a heated mold using ethylene oxide-capped poly(propylene oxide) polyols. The isocyanate component can comprise of methylene diphenyl diisocyanate (MDI) derivatives, toluene diisocyanate (TDI), or a mixture thereof. EoL molded HR flexible polyurethane foam is characterized by having a resiliency value of exceeding 50% as measured using a ball rebound test (ASTM D3574). Exemplary articles in which EoL molded HR flexible polyurethane foam can be sourced from include automotive seats and the seating foam of furniture.

By EoL conventional flexible polyurethane foam we refer here to foam manufactured for instance by using the slabstock production process, and characterized by being based on polyols that present mainly secondary OH groups. Additionally, foam of this class is prepared solely using toluene diisocyanate as the isocyanate component. EoL conventional flexible polyurethane foam is characterized by having a resiliency value of less than 50% as measured using a ball rebound test (ASTM D3574). Exemplary articles in which EoL conventional flexible polyurethane foam can be sourced from include mattresses.

As discussed in U.S. Publication No. 2023/0399486, polyurethane EoL material includes both post-production trims and off-specification materials, and post-consumer EoL material, e.g., in the form of scrap furniture, pillows, cushions, mattresses, car seats and shoe soles. The polyurethane EoL material may, e.g., contain fillers and/or additives. The polyurethane EoL material may be, e.g., solid or foamed. There are no restrictions in the process according to exemplary embodiment in terms of the type and composition of the polyurethane EoL material. Further, it may not be necessary to provide polyurethane EoL material of a single type, e.g., it may be advantageously possible to dispense with an expensive step of pre-sorting the polyurethane EoL material. For technical reasons, it is in fact common to separate extraneous matter such as textiles, steel, wood and other extraneous substances from the polyurethane EoL material.

The process according to embodiments is suitable for processing polyurethane EoL foam, e.g., for processing flexible polyurethane foam, cellular and microcellular polyurethane materials, polyurethane elastomers, PUR integral rigid foam, semi-rigid polyurethanes, thermoplastic polyurethanes (TPU), rigid polyurethane foams and rigid PUR/PIR foams. The polyurethane EoL material can be processed in sorted or un-sorted form by means of the process according to the invention. The polyurethane EoL material may originate from production and also from the post-consumer sector. This process according to exemplary embodiments is particularly suitable for recycling a molded HR flexible polyurethane foam (e.g., seating type foam) into a polyol composition especially suitable for the production of HR molded flexible polyurethane foam for automotive uses.

The embodiments seek to address these various concerns by directly generating recycled polyol composition from EoL molded HR flexible polyurethane foam, obtaining a recycled polyol exhibiting a low OH# that is appropriate for reuse in molded HR flexible polyurethane foams.

The final recycled polyol dispersion is useful for making molded HR flexible foam. Embodiments relate to an isocyanate reactive recycled polyol dispersion from the reaction of a mixture which includes the following:

i. at least one molded HR flexible polyurethane EoL foam that is adequate for automotive seating foam properties such as density of higher than 30 g/L, resiliency of higher than 50%, and compression load deflection (CLD) from 2 kPa to 20kPa at room temperature, where room temperature and the compression load deflection is defined by the ISO 3386-1 standard.

ii. at least one polycarboxylic acid or polycarboxylic acid derivative (e.g. an anhydride) at an amount of >10 wt% and <15 wt%

iii. at least one ethylene oxide (EO)-capped propylene oxide polyether polyol having a primary hydroxyl group content of greater than 50%, such as in a range of 50% to 100%, and an average hydroxyl number from 20 mg KOH/g to 40 mg KOH/g

iv. at least one polyalkylene glycol HO-CH(R)CH_2($OCH_2CH(R)$)$_n$-OH where R = H or (CH_3) and n ranges from 1 to 10. (such as dipropylene glycol)

v. at least one polyalkylene glycol that is different from iii and iv where its average functionality, f, ranging from 1.9 to 6.0, (such as OH# = 40 – 60 mg KOH/g)

wherein the recycled polyol composition has

- average hydroxyl number OH# ranging from 30 to 50 mg KOH/g (ASTM E1899-16),

- average viscosity values ranging from 5 to 15 Pa.s at 25°C (Brookfield DV-II+ Cone and Plate Viscometer).

Referring to the above, a further description of the materials is provided in Table 1 and the weight percent is based on a total weight of the mixture.

Table 1

Material	Physical form	Lower Limit	Upper Limit
An EoL molded HR flexible polyurethane foam	Solid	10 wt%	50 wt%
An EO-capped Poly(propylene oxide)polyol of average OH# 20-40 mg KOH/g having a primary alcohol content greater than 50%	Liquid	10 wt%	30 wt%
A conventional polyether polyol having an average OH# 40-60 mg KOH/g having a primary alcohol content less than 20%	Liquid	10 wt%	30 wt%
A dicarboxylic acid or dicarboxylic acid derivative	Liquid	10 wt%	15 wt%
A short chain polyether diol	Liquid	0.1 wt%	5 wt%

The mixture may further include from 1 wt% to 5 wt%, based on a total weight of the mixture, a hydrogen peroxide solution. The solution may include from 20 wt% to 50 wt% of hydrogen peroxide, based on a total weight of the hydrogen peroxide solution. The remaining portion of the solution may be water and/or other solvents.

The isocyanate reactive recycled polyol composition may include one or more polyether polyols prepared by polyaddition of alkylene oxides such as propylene oxide and/or ethylene oxide onto polyhydroxy functional starter compounds in the presence of catalysts known in the art. Polyether polyols may be prepared from a starter compound and one or more alkylene oxides, for example, ethylene oxide, propylene oxide, and/or butylene oxide. Starter compounds may include, but are not limited to, molecules having 1 to 8 hydroxyl groups per molecule, such as ethylene glycol, propylene glycol, diethylene glycol, dipropylene glycol, triethylene glycol, tripropylene glycol, 1,4-butanediol, 1,6-hexanediol, triethanolamine, diethanolamine, diisopropanolamine, bisphenol A, glycerol, diglycerol, triglycerol, trimethylolpropane, di(trimethylolpropane) pentaerythritol, dipentaerythritol, tripentaerythritol, sugars and sugar alcohols such as sucrose and sorbitol, and the like. Polyether polyols may also include polyols reacted with polyethers formed from copolymers of alkylene oxides, including block copolymers and polyethers "capped" with hydroxyethyl and/or hydroxypropyl oligomers or polymers.

The EO-capped poly(propylene oxide) polyol disclosed herein comprises a block copolymer in which a poly(propylene oxide) polyol is reacted with ethylene oxide to form an ethylene oxide cap. The EO-capped poly(propylene oxide) polyol disclosed herein may have an average hydroxyl number (or OH number) as determined according to ASTM D4274 ranging from 15 mg KOH/g to 50 mg KOH/g, 20 mg KOH/g to 40 mg KOH/g, or 25 mg KOH/g to 35 mg KOH/g. The EO-capped poly(propylene oxide) polyol disclosed herein may have a primary hydroxyl content that exceeds 50% according to ASTM D4273.

The conventional polyether polyol disclosed herein is either a homopolymer of alkylene oxides or a random copolymer of alkylene oxides. The conventional polyether polyol disclosed herein may have an average hydroxyl number (or OH number) as determined according to ASTM D4274 ranging from 35 mg KOH/g to 70 mg KOH/g, 40 mg KOH/g to 65 mg KOH/g, or 45 mg KOH/g to 60 mg KOH/g. The conventional polyether polyol disclosed herein may have a primary hydroxyl content below 20%, such as in a range of 0% to 20%, according to ASTM D4273.

Examples of the dicarboxylic acid or derivatives thereof include the following that were used in the examples below maleic acid, phthalic acid, homophthalic acid, succinic acid, glutaric acid, 2-methyl glutaric acid, 3-methyl glutaric acid, phthalic anhydride, maleic anhydride, succinic anhydride, adipic acid, or a combination of two or more thereof.

Examples of the short chain polyether diol that were used in the examples below include the following dipropylene glycol, tripropylene glycol, diethylene glycol, triethylene glycol or a combination of two or more thereof.

In some cases, methods may include generating a recycled polyol composition containing polyols released from a polyurethane EoL material that include: a) combining, at room temperature, to form a mixture:at least one ethylene oxide (EO)-capped poly(propylene oxide) polyether polyol having a primary hydroxyl content greater than 50% and an average hydroxyl number from 20 mg KOH/g to 40 mg KOH/g, at least one dicarboxylic acid or dicarboxylic acid derivative, a hydrogen peroxide solution, and optionally at least one at polyether polyol having a primary hydroxyl group content of less than 20% and an average hydroxyl number from 40 mg KOH/g to 60 mg KOH/g; b) heating the mixture to 120°C, adding acrylic acid, and heating the mixture to a temperature in the range of 160 °C to 190°C; c) adding the polyurethane EoL material to the mixture to the at least partially depolymerize; d) heating the mixture to a temperature ranging from 200 °C to 230°C and adding at least one dicarboxylic acid or dicarboxylic acid derivative to produce the recycled polyol composition , and e) cooling the mixture to a temperature in a range of 180 °C to 200°C'and adding a short chain polyether diol, and optionally adding a second portion of adding at least one dicarboxylic acid or dicarboxylic acid derivative.

The recycled polyol composition may be further reacted with a polyisocyanate to form a new molded HR flexible polyurethane foam. Similarly, the resultant new foam may have a density of higher than 30 g/L, resiliency of higher than 50%, and compression load deflection (CLD) of from 2 kPa to 20 kPa at room temperature. This recycled polyol composition can be optionally further blended with additional polyols, such as those typically used to make molded HR polyurethane foams for automotive uses, prior to forming the new foam. For example, the recycled polyol composition may be blended with at least one block copolymer including a poly(propylene oxide) derived backbone and ethylene oxide derived capping (e.g., from 2 wt% to 30 wt% of EO cap). See for example the Inventive example 2 in Table 4 below.

Recycled polyol compositions may be useful as feedstocks for various processes for a number of industrial and automotive applications, including the production of molded foams for seating, insulation, and sound dampening.

Examples

The following examples are provided to illustrate the embodiments of the invention, but are not intended to limit the scope thereof. All parts and percentages are by weight unless otherwise indicated. All molecular weight is number average molecular weight unless otherwise indicated.

The following materials listed in Table 2 are used in the examples.

Table 2

Name	Ingredient type	Chemical Description	Source
Polyol 1	Polyol	High functionality (4.7), average OH number = 32.5 mg KOH/g, EO-capped poly(propylene oxide) polyol	The Dow Chemical Company
Polyol 2	Polyol	Styrene acrylonitrile based copolymer polyether polyol, average OH number = 22 mg KOH/g, 40% solids	The Dow Chemical Company
Polyol 3	Polyol	Glycerin initiated, average OH number = 33.5 mg KOH/g, high ethylene oxide content polyether polyol	The Dow Chemical Company
Polyol 4	Polyol	Glycerin initiated, average OH number = 27 mg KOH/g, EO-capped poly(propylene oxide) polyol	The Dow Chemical Company

Polyol 5	Polyol	Glycerin-initiated, average OH number = 56.5 mg KOH/g, all PO,	The Dow Chemical Company
Polyol 6	Polyol	Glycerine initated, average OH number = 29 mg KOH/g, EO-capped poly(propylene oxide) polyol	The Dow Chemical Company
EoL Mattress Foam	Foam	Shredded, end-of-life conventional flexible polyurethane foam collected from mattress articles by a municipal supplier	--
EoL Automotive Foam	Foam	Shredded, end-of-life high resilience flexible polyurethane foam collected from automotive vehicle seats by a recycling company	--
DEA	Catalyst-crosslinker	Diethanolamine	TCI / Aldrich
DABCO® NE1070 Amine Catalyst	Catalyst	3-(dimethylamino)propylurea 1,3-bis[3-(dimethylamino)propyl]urea Catalyst	Evonik Industries
Water	Blowing agent	Chemical blowing agent for foam	--
POLYCAT® 15	Catalyst	Bis (N,N-dimethyl-3-amino-propyl) amine. Catalyst	Evonik Industries
Niax™ DMEE	Catalyst	2-[2-(Dimethylamino)ethoxy]ethanol ammine Catalyst	Momentive
Hydrogen Peroxide 30% in water	Reagent	Foam digestion reagent	Millipore Sigma
Phthalic anhydride	Reagnt	Dicarboxylic acid derivative for foam digestion	TCI / Aldrich
Maleic anhydride	Reagent	Dicarboxylic acid derivative for foam digestion	TCI / Aldrich
Dipropylene glycol	Glycol		TCI / Aldrich
Acrylic acid		Foam digestion reagent	TCI / Aldrich
TEGOSTAB® B 8734 LF 2	Surfactant	Silicone Surfactant	Evonik Industries
Isocyanate 1	Isocyanate	Blend of MDI Isomers chain extended with triol 2500 EW	The Dow Chemical Company

Referring to the above, Isocyanate 1 is prepared as follows: load in the reactor about 39 parts of pure MDI consisting of 70 % 4,4'-MDI and 30 % 2,4'-MDI, about 16.76 parts pure MDI consisting of 50 % 4,4'-MDI and 50 % 2,4'-MDI, about 38 parts polymeric MDI with viscosity about 220 mPas at 25°C, bring the mix to about 70°C and under stirring add about 1 part silicone surfactant, and about 5 parts of glycerine initiated EO/PO mixfeed polyol with 60 % EO content and OH number about 22 mg KOH/g.

Recycled Polyol Preparation (1000 gram scale total charge of raw materials). Digesting polyols are loaded into the reaction vessel, equipped with mechanical stirrer, nitrogen inlet, condenser, and a spare neck for material addition, at room temperature under inert atmosphere, followed by anhydride and peroxide while mixing for 15 minutes. This mixture is slowly heated up to about 190°C. When the temperature of the mixture reaches about 120°C, acrylic acid is added. In the 4th step, when the temperature reaches about 160-165°C, a shredded PU foam (about 2cm size irregular shape pieces) are loaded into the reaction mixture gradually while ensuring the 1st portion is dispersed and wetted sufficiently before the 2nd portion is dosed, , providing efficient mixing. Once the entirety of the foam is loaded and dispersed, the temperature is increased to 215°C and let the mixture to depolymerize while being stirred for 2.5 hours. Next, a portion of the maleic anhydride is dosed and the temperature is increased to 225°C and let to react for another 1h. The mixture is cooled down to 200°C, added DPG and the rest of the maleic anhydride, and stirred for another 15 minutes before cooling down to below 100°C for filtration.

Process for Foam Preparation. Foam samples were made using a thermostatic aluminum mold with dimensions of 25x25x8 cm and volume of 5 liters. Foams were prepared with 75g/l of density and using a mold temperature of 50°C. A demolding time of 5 minutes ensured proper demolding. A hand mix procedure was followed to prepare the aforementioned foams. The polyols, surfactants, catalyst and water were pre-weighted in a 1-liter plastic cup and mixed using an automatic Heidolph stirrer with a speed of 3000 rpm for 1 minute. The appropriate amount of Isocyanate, stored in a different 1-liter cup, was poured on top of the Polyol mixture. This final mixture was then mixed for 10 seconds with a speed of 3000rpm and then poured inside the 25x25x8cm mold. After 5 minutes the foam was taken out of the mold.

Examples Demonstrating Improved Polyol Processability, in the final reaction with isocyanates to give molded HR flexible polyurethane foam, by Increasing Phthalic Anhydride relative content. See Table 3.

Table 3

Recycled Polyol Formulations		**Comparative Example 1**	**Comparative Example 2**	**Comparative Example 3**	**Inventive Example 1**
Polyol 4		None	16.50%	15.80%	15.80%
Polyol 5		36.00%	18.00%	18.00%	18.50%
Phthalic Anhydride		9.00%	10.50%	11.20%	11.20%
Hydrogen Peroxide 30%		2.00%	2.00%	2.00%	1.50%
Acrylic Acid		1.00%	1.00%	1.00%	1.00%
EoL Mattress Foam		40.50%	40.50%	40.50%	None
EoL Automotive Foam		None	None	None	40.50%
Maleic Anhydride		1.00%	1.00%	1.00%	1.00%
DPG (Dipropylene Glycol)		0.50%	0.50%	0.50%	0.50%
Polyol 6		10.00%	10.00%	10.00%	10.00%
Recycled Polyol Properties	(desired)				
OH# as KOH(mg/g)	25-45	55	49	46.2	30.2
Viscosity at 25°C (mPa.s)	<16000	7000	12700	8927	14090
Acid number (mg/g)	< 3	2	2.7	2.9	2.2
Foam addition time	Less time	2h 15 min	2 h 18 min	3 h	2 h 20 min

In Table 3 are the analytical results of the recycled polyols produced according to the above. Full formulation and analytical results are also compiled in Table 3 above. Comparative Example 1 was produced with 9% phthalic anhydride (PA). The foam addition took about 2h 15min (Comparative Example-1). Next the same procedure was followed by increasing the amount of PA (10.5%) and using high MW EO-capped polyol (Comparative Example-2) in which foam addition was 2h 18min. The PA was next increased to 11.2%, and the foam addition took 3h (Comparative Example-3). Finally, EoL automotive foams were digested with 11.2% PA and high MW EO-capped polyol (Inventive Example-1). Here foam addition took less time, 2h 20min. Foam addition time is related to the speed of foam degradation via chemical reaction, therefore digestion of the automotive foam was surprisingly faster than conventional foam. The usefulness of these recycled polyol products were tested in molded HR automotive seating polyurethane foam production.

Table 4

Foam Recipes	**Comparative Example 4**	**Comparative Example 5**	**Comparative Example 6**	**Inventive Example 2**
Ingredient	**wt%**	**wt%**	**wt%**	**wt%**
Polyol 4	43.66	23.66	23.66	23.66
Polyol 1	18.01	18.01	18.01	18.01
Polyol 2	30.01	30.01	30.01	30.01
Polyol 3	3.9	3.9	3.9	3.9
DEA	0.24	0.24	0.24	0.24
Dabco NE1070	0.95	0.95	0.95	0.95
SH WATER	2.33	2.33	2.33	2.33
POLYCAT 15	0.3	0.3	0.3	0.3
Niax* DMEE	0.1	0.1	0.1	0.1
Tegostab B 8734 LF 2	0.5	0.5	0.5	0.5
Recycled Polyol: Comparative Example 1	-	**20**	-	-
Recycled Polyol: Comparative Example 3	-	-	**20**	-
Recycled Polyol: Inventive Example 1	-	-	-	**20**
Total	100	100	100	100
Foam prepared by reaction with Isocyanate 1	Index 85	Index 85	Index 85	Index 85
Foam Properties	**Comp. Ex. 4**	**Comp. Ex. 5**	**Comp. Ex. 6**	**Inv. Ex. 2**
Cell size [µm]	500	1045	791	529
Air Flow [dm3/s]	0.28	0.1	0.03	0.51
Flowability Rating	3	1	2	3
Tear Strength (N/mm)	0.24	0.23	0.24	0.31
Resiliency (%)	59	30	32	54

The HR automotive seating foams were prepared using the recycled polyols incorporated in the isocyanate reactive blend in 20 parts and as a replacement of one of the virgin polyols. Exemplary formulation and, properties of the foams prepared starting from them are, reported in Table 4. Comparative Example 4 refers to the reference foam formulation, in which no recycled polyol was used. Comparative Example 5 refers to a foam formulation in which recycled polyol (Comparative Example 1) was used. Comparative Example 6 refers to a foam formulation in which a recycled polyol made with EO-capped polyol and EoL conventional flexible polyurethane foam (Comparative Example 3) was used. Inventive Example 2 refers to a foam formulation incorporating the inventive recycled polyol, Inventive Example 1, made from the digestion of EoL molded HR flexible polyurethane foam with a recipe that includes high MW EO-capped polyol Inventive Example 2 is able to match the physical mechanical properties of the Comparative Example 4, that does not contain any recycled polyol and that is designed to have optimal properties as required for molded automotive seating foam applications.

In particular, the physical mechanical properties of the Inventive Example-2 are surprisingly matching the ones obtained in the Comparative Example-4 where no recycled polyol is used. The reported data are showing a decrease of the performance in the Comparative Example-5 and-6, the cells are large in diameter, and at the same time the air flow of foams is decreased. Furthermore, a poorer filling of the mold by the reactive mixture is detected. When using the innovative recipe of the Inventive Example-1 to produce the foam of Inventive Example-2 it is surprisingly possible to achieve the same properties found when no recycled polyol is used. This enables meeting the sought after properties requested by the application.

Test methods are as follows:

Hydroxyl number is measured according to ASTM E1899-16.

Acid number is measured by potentiometric titration with standardized 0.01N potassium hydroxide solution according to ASTM D7253.

Viscosity is measured on a Brookfield DV-II+ Cone and Plate Viscometer with cone CPE-52 at 25 °C at speed/rpm that yields torque reading between 10 and 90% of range.

Water content is measured by Karl Fisher coulometric titration on Mettler DL39 Coulometric Karl Fisher Titrator according to ASTM D4672 and ASTME1064.

Air Flow Measurement is measured according to ASTM D-3574 with a FX 3300 LabAir System.

Cell size is measured through a Goldlücke PORE!SCAN optical microscope instrument with automatic imaging analysis and cell size determination.

Tear Strength is measured according to the testing method PV3410 with a 100 mm/min speed in an INSTRON 5566 dynamometer.

Compression Load Deflection (CLD) is measured according to ISO 3386-1. The reported value is the $CV_{40}\%$.

Resiliency is measured according to ASTM D3574.

Density is measured according to ISO 845.

Flowability or Flowability Rating, is assessed using a standard mold according to an internal test method. The comparative test is performed on the same mold, under the same conditions and casting in the mold the same amount of reactive mixture.

The capacity of filling the mold is compared between foams at the very same amount of reactive mixture (isocyanate-reactive component plus isocyanate) cast in a certain mold. A rating from 1 to 4 is given as following:

1. The mold is not filled;
2. The mold is filled but with defects. Defects are voids (free of material) in the foam with a diameter of greater than 5mm. Defects for rating 2 are observed across the entire thickness of the molded foam.
3. The mold is filled but with defects only on about half of the thickness of the molded foam.
4. The mold is filled and no defect is detected.

Note that in the test result, the Comparative Example-4 is not achieving the highest rating (flowability rating for Comparative Example-4 is only 3) because the test is designed to highlight defects in flowability, hence the amount of reacting mixture added into the mold is barely sufficient to fill the mold, and the defects appear.

While the foregoing is directed to exemplary embodiments, other and further embodiments may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

Listing of the Claims:

1. A recycled polyol composition containing polyols released from polyurethane EoL material, made by the process of:

providing the polyurethane EoL material; and

forming a reaction mixture by mixing from 10 wt% to 50 wt%, based on the total weight of the reaction mixture, of polyurethane EoL material with the following:

from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of at least one ethylene oxide (EO)-capped poly(propylene oxide) polyether polyol having a primary hydroxyl content greater than 50% and an average hydroxyl number from 20 mg KOH/g to 40 mg KOH/g,

from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of at least one polyether polyol having a primary hydroxyl group content of less than 20% and an average hydroxyl number from 40 mg KOH/g to 60 mg KOH/g,

from 10 wt% to 15 wt%, based on the total weight of the reaction mixture, of at least one dicarboxylic acid or dicarboxylic acid derivative, and

from 0.1 wt% to 5 wt%, based on the total weight of the reaction mixture, of at least one short chain polyether diol that is different from the at least one first polyether polyol and the at least one second polyether polyol,

the reaction mixture forming the recycled polyol composition, which includes polyols released from the polyurethane EoL material, has an average hydroxyl number from 30 mg KOH/g to 50 mg KOH/g, and has an average viscosity from 5 Pa·s @ 25°C to 15 Pa·s @ 25°C.

3.The composition according to claim 1, wherein the polyurethane EoL material is molded high resilience flexible polyurethane foam having a density of higher than 30 g/L, resiliency of higher than 50%, and compression load deflection (CLD) of from 2 kPa to 20 kPa at room temperature.

4. The composition according to claim 1 or claim 2, further including reacting at least the recycled polyol composition with a polyisocyanate to form a new molded high resilience flexible polyurethane foam having a density of higher than 30 g/L, resiliency of higher than 50%, and compression load deflection (CLD) of from 2 kPa to 20 kPa at room temperature.

5. The composition according to any one of claims 1 to 3, wherein the reaction mixture further includes from 1 wt% to 5 wt%, based on a total weight of the reaction mixture, of a hydrogen peroxide solution that includes from 20 wt% to 50 wt% of hydrogen peroxide in water, based on a total weight of the hydrogen peroxide solution, and of acrylic acid.

6. The composition according to any one of claims 1 to 4, wherein the reaction mixture forming the recycled polyol composition, is a recycled polyol dispersion.

7. A method of generating a recycled polyol composition containing polyols released from polyurethane EoL material, comprising:

forming a reaction mixture comprising a polyurethane EoL material at 10 wt% to 50 wt%, based on the total weight of the reaction mixture, and the following:

from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of at least one ethylene oxide (EO)-capped poly(propylene oxide) polyether polyol having a primary hydroxyl content greater than 50% and an average hydroxyl number from 20 mg KOH/g to 40 mg KOH/g,

from 10 wt% to 30 wt%, based on the total weight of the reaction mixture, of at least one polyether polyol having a primary hydroxyl group content of less than 20% and an average hydroxyl number from 40 mg KOH/g to 60 mg KOH/g,

from10 wt% to 15 wt%, based on the total weight of the reaction mixture, of at least one dicarboxylic acid or dicarboxylic acid derivative, and

from 0.1 wt% to 5 wt%, based on the total weight of the reaction mixture, of at least one short chain polyether diol that is different from the at least one first polyether polyol and the at least one second polyether polyol,

wherein the reaction mixture forming the recycled polyol composition, comprising polyols released from the polyurethane EoL material, has an average hydroxyl number from 30 mg KOH/g to 50 mg KOH/g, and has an average viscosity from 5 Pa·s @ 25°C to 15 Pa·s @ 25°C.

8. A method of generating a recycled polyol composition containing polyols released from a polyurethane EoL material, comprising:

a) combining, at room temperature, to form a mixture:

at least one ethylene oxide (EO)-capped poly(propylene oxide) polyether polyol having a primary hydroxyl content greater than 50% and an average hydroxyl number from 20 mg KOH/g to 40 mg KOH/g,

at least one dicarboxylic acid or dicarboxylic acid derivative,

a hydrogen peroxide solution, and

optionally at least one at polyether polyol having a primary hydroxyl group content of less than 20% and an average hydroxyl number from 40 mg KOH/g to 60 mg KOH/g

b) heating the mixture to 120 °C, adding acrylic acid, and heating the mixture to a temperature in the range of 160 °C to 190 °C

c) adding the polyurethane EoL material to the mixture to the at least partially depolymerize,

d) heating the mixture to a temperature ranging from 200 °C to 230 °C and adding at least one dicarboxylic acid or dicarboxylic acid derivative to produce the recycled polyol composition, and

e) cooling the mixture to a temperature in a range of 180 °C to 200 °C and adding a short chain polyether diol.

9. The method of claim 8, wherein e) further comprises adding a second amount of dicarboxylic acid or dicarboxylic acid derivative.