



A Process for Producing Improved Palm-based Flexible Polyurethane Foams

Field of Invention

- 5 The present invention relates to a process for producing improved palm-based flexible polyurethane foams (hereinafter referred to as PUFs) by incorporating phthalates as additive to improve cross-linking within the polymer structure.

Background of the Invention

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The production of polyurethane foams is effected partly continuously by free foaming is so-called "blocks" and partly in closed molds, whereby, depending on the composition of the polyurethane mixtures, open-pored or close-pored foams with or without a closed outer skin are formed.

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- A wide range of foam materials containing urethane groups and having different physical properties has long been produced on an industrial scale. Conventional flexible foams are made by reacting a polyisocyanate and a relatively high molecular weight isocyanate reactive polymer, often a polyester or polyether polyol, in the presence of a blowing agent and optionally further using limited amounts of relatively low molecular weight chain extenders and cross-linkers and optionally using additives like catalysts, surfactants, fire retardants, stabilizers and antioxidants. The relatively high molecular weight isocyanate reactive polymer in general represents the highest weight fraction of the foam. Such flexible foams may be prepared according to the one-shot, the quasi- or semi-prepolymer or the prepolymer process. Such flexible foams may be molded foams or slab stock foams and may be used as cushioning material in furniture and automotive seating and in mattresses, as carpet backing, as hydrophilic foam in diapers and as packaging foam. Further they may be used for acoustic applications, e.g. sound insulation. Examples of prior art for these conventional flexible foams are EP-10850, EP-22617, EP-111121, EP-296449, EP-309217, EP-309218, EP-392788 and EP-442631.
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The release of the foams formed from the mold raises special problems in the case of



polyurethane foams, because the low-molecular-weight isocyanates used for their production, which are known as adhesives for many purposes, can come easily in contact with the mold wall. The difficulties in mold release, which already exist in casting polyurethane, are considerably increased in polyurethane foam, due to the
5 nature of this material.

These difficulties cannot be satisfactorily eliminated today, even by the use of relatively large amounts of mold release agents. The conventional mold release agents consist preferably of solutions of hydrocarbon waxes, partly in mixture with silicones,
10 in benzene and similar solvents. In some cases it is necessary to apply wax-like pastes on the mold wall, and to polish them in order to smooth the surface. The method, however, cannot be used particularly for the mold release agent, because the fine unevenness of the mold surface that is to be reproduced in the foam, levels the production of integral skin foams.

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The present invention relates to compositions for preparing water blown flexible polyurethane foam by reacting palm-based polyols with isocyanate by adding a cross linker selected from alkyl aryl phthalates and dialkyl phthalates to the reaction compounds. Water is used as a chemical blowing agent, which releases carbon dioxide
20 to foam the polyurethane composition.

Phthalates group especially alkyl aryl phthalates and dialkyl phthalates was described in few patents, US5624968, W0007_3368 and G13890280, as plasticizer to improve the softness and flexibility of flexible PUFs.

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Besides polyurethane, phthalates based on n-butyl alcohol or beta-ethylhexyl alcohol was used as plasticizer in the polymer composition for shoe plasticized material.

In Japanese patent application No. 59-226034, published December 19, 1984, a method
30 for manufacturing a foamed urethane molded article is described. In the invention, a phthalic acid plasticizer, such as di-2-ethylhexyl phthalate, butyl benzyl phthalate and dibutyl phthalate was added to the reaction compounds, which consist of polyol, isocyanate, a fluorocarbon compound and water to lower mold temperature required for

molding the desired materials specifically based on rigid foams.

The objective of the present invention is to provide a new method of producing improved palm-based flexible polyurethane foams (PUFs) by incorporating phthalates as additive to improve cross-linking within the polymer structure.

Another objective of the present invention is to describe the compositions for preparing water blown flexible polyurethane foam by reacting palm-based polyols with isocyanate by adding a cross linker selected from alkyl aryl phthalates and dialkyl phthalates to the reaction compounds.

Summary of the Invention

The objective of the present invention is to provide a new method of producing improved palm-based flexible polyurethane foams (PUFs) by incorporating phthalates as additive to improve cross-linking within the polymer structure.

Another objective of the present invention is to describe the compositions for preparing water blown flexible polyurethane foam by reacting palm-based polyols with isocyanate by adding a cross linker selected from alkyl aryl phthalates and dialkyl phthalates to the reaction compounds.

The present invention describes the production and compositions of an improved palm-based water blown flexible polyurethane foams (PUFs) by incorporating phthalates into the polyurethane formulation as a cross linker.

A process for producing flexible polyurethane foams (PUFs) according to the present invention comprising the steps:

- a). preparing of polyols;
- b). mixing of said polyols with additives including blowing agent and phthalate as cross linker to improve the cross linking within the polymer structure; and
- c). mixing of mixture obtained from step (b) with an isocyanate to form polyurethane foam.

Polyols is the main raw material for the production of polyurethane foams, which may

be an aliphatic or aromatic polyhydroxy compound, which will react with the isocyanate. The process for producing the polyol has been disclosed in Malaysia Patent No. MY-114189-A, Patent Singapore: 55223 and Patent Application/Indonesia: P962884. This disclosure is incorporated herein as reference.

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The additive is a combination of phthalates as cross linker, silicone surfactant, amine catalyst, tin catalyst, flame retardant and chain extender. Phthalates, in the amount 0.5 to 5.0 pphp (parts per hundred polyols) is incorporated into the formulation to increase the cross linking within the polymer network. The phthalates are selected from the
10 group of alkyl aryl phthalates and dialkyl phthalates. Water is used as a chemical blowing agent, which releases carbon dioxide to foam the polyurethane composition. Preferably, the water used in the present invention is about 3 to 5 pphp.

In the preferred embodiment of the present invention, isocyanate is any one or
15 combination of toluene diisocyanate (TDI), diphenylmethane diisocyanate (MDI) or its derivatives and more preferably modified MDI containing mixture of modified toluene diisocyanate and modified diphenylmethane diisocyanate. The modified MDI is preferably 50 to 100 pphp.

20 **Brief Description of the Drawings**

Figure 1 shows the table of characteristics of palm-based polyols.

Detailed Descriptions of the Preferred Embodiments

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A detailed description of the preferred embodiment of the present invention will now be provided.

The present invention describes the production and compositions of an improved
30 palm-based flexible polyurethane foams (PUFs) by incorporating phthalates into the polyurethane formulation as a cross linker.

A process for producing flexible polyurethane foams (PUFs) comprising the steps:

- a). preparing of polyols;
- b). b). mixing of said polyols with additives including blowing agent and phthalate as cross linker to improve the cross linking within the polymer structure; and
- c). mixing of mixture obtained from step (b) with an isocyanate to form polyurethane foam.

The process for producing of the polyol has been disclosed in Malaysia Patent No. MY-114189-A, Patent Singapore: 55223 and Patent Application/Indonesia: P962884. This disclosure is incorporated herein as reference.

The blowing agent used in the present invention is preferably water. Other blowing agent such as methylene chloride may also be used, either in place of or in combination with water.

First step involving of preparing of polyols. Process of preparing polyols involves a few steps and can be referred to Patent Malaysia: MY-114189-A, Patent Singapore: 55223 and Patent Application Indonesia: P962884. There are four types of polyols that can be produced from this patented process. The characteristics of these polyols are illustrated in Figure 1.

Additives and blowing agent are then added into the mixture and mixed thoroughly until a homogenous mixture obtained. The mixing is done at about 2000 rpm until the mixture turns creamy. The additive is a combination of phthalate as cross linker, silicone surfactant, amine catalyst, tin catalyst, flame retardant and chain extender. The functions of these additives are to control and modify the final polyurethane polymer, in which phthalate, in the amount 0.5 to 5.0 pphp (parts per hundred polyols) is incorporated into the formulation to increase the cross linking within the polymer network. The phthalates are selected from the group of alkyl aryl phthalates and dialkyl phthalates. Another main ingredient in polyurethane formulation is blowing agent. In this process, about 3 to 5 pphp of water is used as chemical blowing agent, which reacts with isocyanate to form unstable carbamic acid, which decomposes to urea and carbon dioxide. Carbon dioxide provides the expansion or generates the cell structure of foam.

Next, mixing of the blended polyols obtained from the above with an isocyanate to form polyurethane foam. The isocyanate may be selected from the group of toluene diisocyanate (TDI), diphenylmethane diisocyanate (MDI) or its derivatives. The preferred isocyanate for flexible PUFs production according to the present invention is modified MDI, which contain mixture of modified toluene diisocyanate and modified diphenylmethane diisocyanate. Preferably the amount of modified MDI is in the range of about 50 to 100 pphp.

Lastly, the prepared polyurethane mixture may be poured into a plastic tray to produce free rise foam. Alternatively, the mixture is poured into a mould to produce molded foam.

The following examples are intended to further illustrate the present invention, without any intent for the present invention to be limited to the specific embodiment described therein.

Example 1: Production of free rise palm-based flexible polyurethane foam

First step: Preparation of free rise palm-based flexible polyurethane foam and testing on its properties.

For the production of free rise flexible foam, about 300g of palm-based polyols (RE-PG3 1) is weighed inside the plastic jug. Then, additives with certain amount such as 3 pphp (parts per hundred polyols) silicone surfactant, 4.5 pphp amine catalyst, 1.5 pphp dimethyl phthalate, 0.6 pphp tin catalyst and 12 pphp water are added into the polyol. The mixture is stirred at 2000 rpm until creamy appearance.

Next, about 210 pphp of modified MDI is added into the polyol blend and mixture is stirred again at about 2000 rpm. The stirring process is stopped when the polyurethane mixture turns creamy. The mixture is poured into a plastic tray and allowed to rise freely. The foaming characteristic such as tack free time is recorded.

Second step: Testing on foam's properties.

The foam is allowed to cure at room temperature for one day before it is cut into test specimen for physical testing. The physical tests are performed according to standard methods:

- 5 (i) Density, where the density is calculated from expression: weight (kg)/volume (m^3)
- (ii) Percentage open-cell content, where this test is conducted by using Micromeritics AccuPyc 1330 Pycnometer. The test is comparable particularly to ASTM D2856.
- (iii) Compressive stress, where this is the hardness at 40% strain from the compression hardness test. The test is conducted according to DIN 53 577.
- 10 (iv) Hysteresis, where it measures the area between loading and unloading curves plotted on the stress vs strain curve during the compression hardness test according to DIN 53577.
- (v) Tear Strength, where this test is conducted according to ASTM 3574 – Method F using the Hounsfield Testing Machine.

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Typical results of the properties of free rise flexible polyurethane foams:

Tack free time (ranging 4-5 minutes), density (ranging 35-45 kg/m^3), percentage open cell content (ranging 97-98%), hysteresis (ranging 50-55%) and tear strength (ranging 90-110 N/m)

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Example 2: Production of palm-based molded flexible polyurethane foam.

First step: Preparation of molded flexible polyurethane foam.

- 25 For the production of molded flexible foam, about 280g of palm-based polyols (RE--PG31) is weighed inside the plastic jug. Then, additives with certain amount such as 2.8 pphp (parts per hundred polyols) silicone surfactant, 5.6 pphp amine catalyst, 1.4 pphp dimethyl phthalate, 1.4 pphp tin catalyst and 11.2 pphp water are added into the polyol. The mixture is stirred at 2000 rpm until creamy appearance.

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About 168 pphp of modified MDI is added into the polyol blend and mixture is stirred again at 2000 rpm. The stirring process is stopped when the polyurethane mixture turns creamy. The mixture is poured into an aluminum mould with dimension 30 cm x 30 cm

x 5 cm and the mould is closed. After 4 minutes, the foam is taken out from the mould and the tack free time is measured.

Second step: Testing on foam's properties.

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The foam is allowed to cure at room temperature before it is cut into test specimen for physical testing. The physical tests are performed according to standard methods:

- (i) Density, where the density is calculated from expression: weight (kg)/volume (m^3).
- 10 (ii) Compressive stress, where this is the hardness at 40% strain from the compression hardness test. This test is conducted according to DIN 53 577.
- (iv) Hysteresis, where it measures the area between loading and unloading curves plotted on the stress vs strain curve during the compression hardness test according to DIN 53577.
- 15 (v) Tear Strength, where this test is conducted according to ASTM 3574 – Method F using the Hounsfield Testing Machine.

Typical results of the properties of molded flexible polyurethane foams:

- Tack free time (ranging 4-5 minutes), density (ranging 45-55 kg/m^3), hysteresis
- 20 (ranging 50-55%), compressive stress (ranging 5-7 kPa), and tear strength (ranging 140-150 N/m).

- It is to be understood that the present invention may be embodied in other specific forms and is not limited to the sole embodiment described above. However
- 25 modification and equivalents of the disclosed concepts such as those which readily occur to one skilled in the art are intended to be included within the scope of the claims which are appended thereto.

Claims

1. A process for producing flexible polyurethane foams (PUFs) comprising the steps:
 - a). preparing of polyols;
 - 5 b). mixing of said polyols with additives including blowing agent and phthalate as cross linker to improve the cross linking within the polymer structure; and
 - c). mixing of mixture obtained from step (b) with an isocyanate to form polyurethane foam.
- 10 2. The process for producing flexible polyurethane foams according to Claim 1, wherein said phthalates are selected from the group of alkyl aryl phthalates or dialkyl phthalates.
3. The process for producing flexible polyurethane foams according to Claim 3,
15 wherein said phthalates is in the range of about 0.5 to 5.0 pphp.
5. The process for producing flexible polyurethane foams according to Claim 1, wherein said additive including phthalate as cross linker, and any one of or a combination of silicone surfactant, amine catalyst, tin catalyst, flame retardant and
20 chain extender.
6. The process for producing flexible polyurethane foams according to Claim 1, wherein said blowing agent is water.
- 25 7. The process for producing flexible polyurethane foams according to Claim 6, wherein said water is in a range of about 3 to 5 pphp.
8. The process for producing flexible polyurethane foams according to Claim 1, wherein said isocyanate is any one or combination of toluene diisocyanate (TDI),
30 diphenylmethane diisocyanate (MDI) or its derivatives.
9. The process for producing flexible polyurethane foams according to Claim 8, wherein said isocyanate is preferably modified MDI containing mixture of modified

toluene diisocyanate and modified diphenylmethane diisocyanate.

10. The process for producing flexible polyurethane foams according to Claim 8, wherein said modified MDI is in the range of about 50 to 100 pphp.

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11. The process for producing flexible polyurethane foams according to Claim 1, wherein it has produced said free rise flexible polyurethane foams with improved properties in term of tack free time of about 4 minutes, density of about 40 kg/m^3 , percentage open-cell of about 98%, hysteresis of about 56% and tear strength of about

10 100 N/m.

12. The process for producing flexible polyurethane foams according to Claim 1, wherein it has produced said molded flexible polyurethane foams with improved properties in term of tack free time of about 4 minutes, hysteresis of about 55 % and

15 tear strength of about 140 N/m.

13. A flexible polyurethane foam produced according to any one of Claims 1 to 12.