

Novel Technology to Influence Hardness of Flexible Polyurethane Foams

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ABSTRACT

Easy, user friendly control of hardness is of high interest for the production of flexible polyurethane foam. There are several ways to influence the hardness. Reduction of hardness and obtaining a softening effect usually has only limited impact on other foam physical properties. However, an increase in hardness is not readily possible without undesirable changes of other foam physical properties in flexible polyurethane foams. There are different ways to increase hardness in flexible polyurethane foams. For example it is possible to strengthen the three dimensional network of the polyurethane foam. Usually a better cross-linking leads to an increased hardness. But normally this also gives rise to a more closed cell structure of the foam. This change in porosity is responsible for an undesired loss of breathability and a different long-term behavior of the polyurethane foam. In order to create a foam hardener which does not affect other foam physical properties, Evonik has put a lot of effort into understanding the hardening effect of foams. This has led to the development of a tailor-made, highly sophisticated foam hardener additive for flexible foam. Therefore we have synthesized a new ORTEGOL[®] product to combine low use level with a significant increase in hardness without impact on other foam physical properties.

INTRODUCTION

Polyurethane products have many uses. The majority of the global consumption of polyurethane products is in the form of foams, with flexible and rigid types being roughly equal in market size. The major markets of flexible polyurethane foam are in the furnishing, packaging and automotive industries [1]. These applications require the foam to have well controlled mechanical properties. Hardness, for instance, is one of the properties which is crucial for these applications. Polyurethanes are formed by the reaction of di- or polyisocyanates with active hydrogen-containing compounds. In water blown systems two hydrogen-containing compounds are used: water and polyols [2]. The cross-linking reaction between isocyanate and polyol leads to urethane groups and is called the gelling reaction (equation 1, figure 1), whereas the reaction between isocyanate and water is called the blowing reaction (equation 2, figure 1). During this reaction instable carbamic acid is generated which afterwards decomposes to the parent amine and carbon dioxide. More than 95% of the volume of the foam is created by the CO₂ built during this reaction [1]. Besides CO₂ the blowing reaction results in the formation of functionalized urea groups. Further reaction of the functionalized urea results in chain extension and incorporation of the resulting urea hard segments into the growing network. A block-segmented copolymer is built up in which the polyol is the soft segment and the urea forms the hard segment [3]. These hard segments contribute to the increase of the hardness of slabstock polyurethane foam.

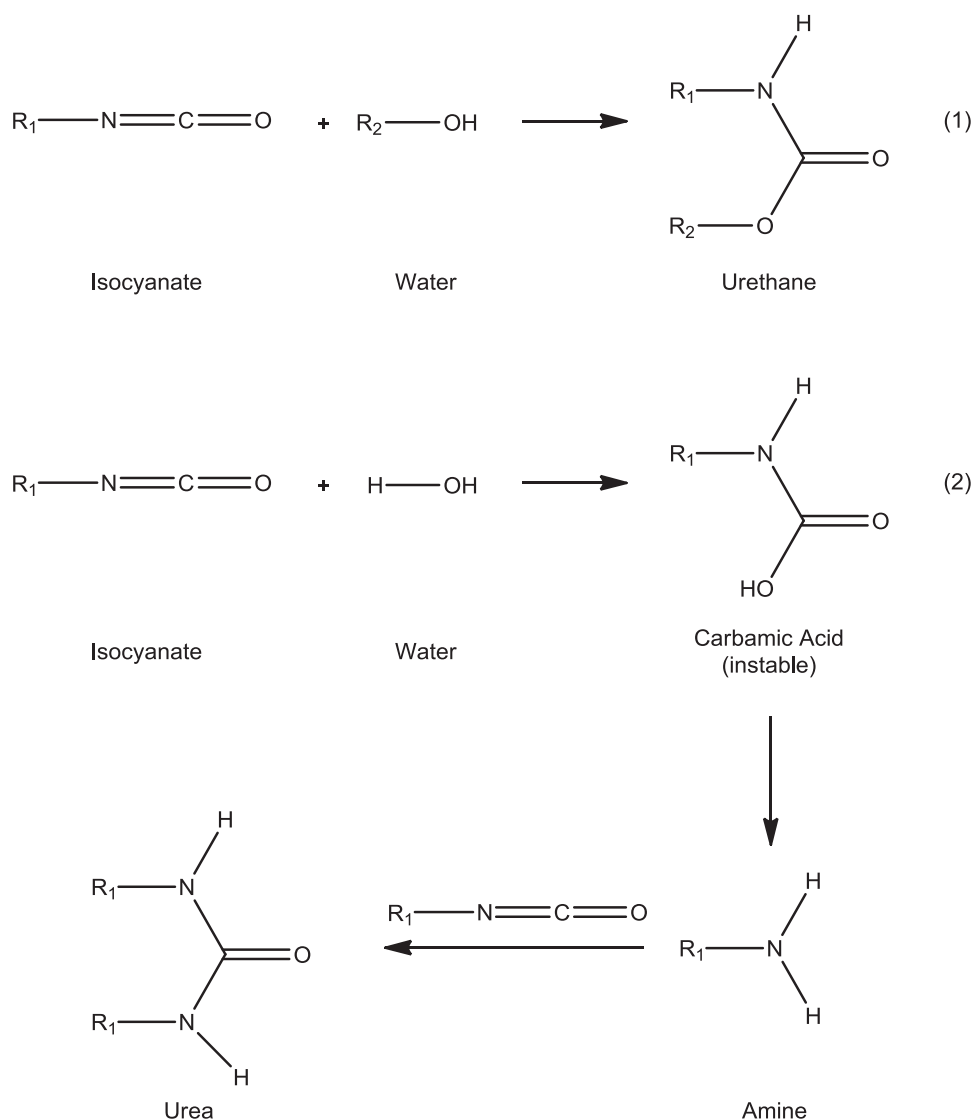


Figure 1. Gelling (1) and blowing (2) reaction during the polyurethane formation.

Control of hardness is one major issue in the production of flexible polyurethane foam. Flexible PU foam must meet diverse requirements depending on various applications. Therefore within the spectrum of flexible polyurethane foams there is a broad range of different grades with different degrees of hardness. Several ways exist in order to influence foam hardness. Reduction of hardness usually is a minor problem. It can either be achieved by the addition of softening additives while keeping the TDI index constant or by reducing the TDI index in combination with an addition of cross-linking additives in order to avoid splits. Generally only limited impact on other foam physical properties is observed. On the other hand an increase of hardness is not readily possible without undesirable changes of other foam physical properties such as air permeability. As already described in former publications [e.g. 1] there are different strategies in order to harden conventional slabstock foam (displayed in table 1). Most of them show serious disadvantages such as an unfavorable influence on the porosity, higher foam costs, an undesirable impact on the emanations of volatile organic compounds (VOCs) or a non-lasting foam hardness which leads to a bad durability of the resulting foam.

| Table 1. Different ways to increase flexible foam hardness. | | | | | |
|---|-----------------------|-------------------------|-------------------|-------------------------|--|
| Method | Influence on porosity | Influence on foam costs | Influence on VOCs | Real or "fake" hardness | Remarks |
| Use of fillers | 0 | 0 | 0 | real | 1) Density adjustment needed 2) Safety issues 3) Foam physical properties suffer |
| Use of polymeric polyol | 0 | +++ | depending | real | |
| Increase of index | ++ | + | 0 | both | Safety issues |
| Increase of tin level | ++ | + | depending | both | |
| Use of cross-linkers | ++ | + | 0 | both | |

Generally it can be observed that higher density foams show higher hardness. For water blown foams an increase of hardness of 0.1 kPa can be obtained when raising the density by 1 kg/m³ or 0.06 pcf (figure 2, left). That is why the addition of fillers also leads to harder foams. This is exemplified in figure 2 (right) with use of CaCO₃ and BaSO₄. However with an increasing amount of filler physical properties like tensile strength and elongation at break suffer which is one major disadvantage. Additionally more cross-linking is needed when using fillers in order to prevent splits.

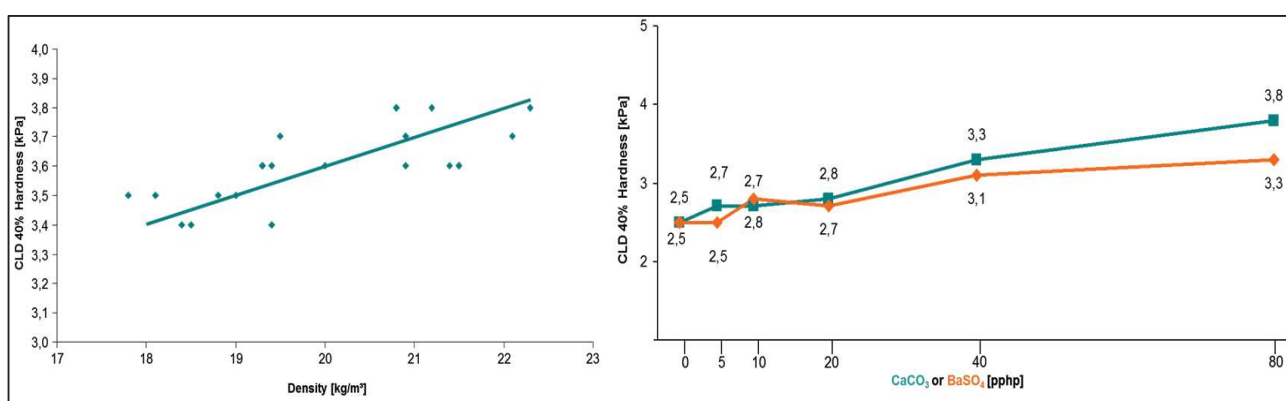


Figure 2. Increase of foam hardness with an increasing density (left) and variation of hardness generated by the addition of mineral fillers without density adjustment (right).

It will be necessary to compensate the density increase if foams of the same density shall be compared. This is either possible by the addition of water or by the addition of methylene chloride. With an increasing amount of water the quantity of aggregated hard segments is enhanced which leads to a harder foam. In contrast a higher methylene chloride loading leads to softer foams because methylene chloride disturbs the aggregation of hard segments. This is also illustrated in figure 3. It has to be noted that an increasing amount of water leads to a higher dosage of TDI (when an identical index is applied). This in turn results in a higher core temperature of the foam and an elevated risk of self-ignition results when the recommended maximum dosage of TDI of 55 pphp is exceeded.

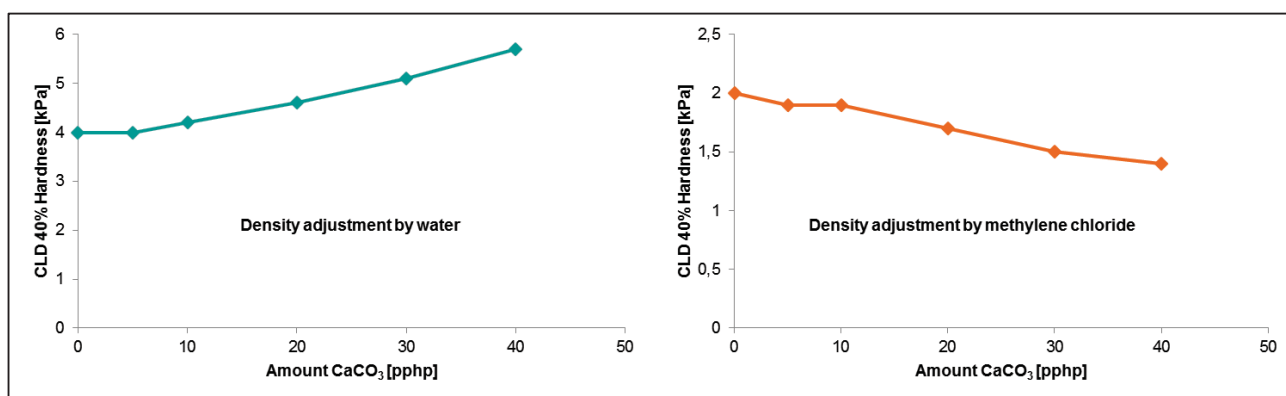


Figure 3. Variation of hardness generated by the addition of mineral filler with density adjustment by water (left) and by methylene chloride (right).

The most widely used method in order to increase the hardness of flexible polyurethane foams is the use of polymeric polyols also known as graft copolymer polyols. Most commonly utilized polymeric polyols are styrene/acrylonitrile (SAN) copolymers and polyurea modified polyols (PHD polyols) which are dispersions of polyurea particles, formed by the reaction between TDI and diamines in a conventional polyol. SAN polyols are obtained through free radical grafting of styrene and acrylonitrile polyether polyols in the presence of a radical initiator like for example AIBN. Polyurethane foams made with a copolymer polyol will have higher hardness than those made with a regular polyol due to the effect of hard organic fillers. However due to the extra step in copolymer polyol manufacturing, the cost of the foam product increases significantly. Additionally, SAN polyols will have a major contribution to the VOC content of flexible foams because rests of styrene and decomposition products of the radical initiator will be present owing to the production method. Another way to increase the hardness of flexible slabstock foams for a given density is to strengthen the polymer network. This can be achieved by several measures: Firstly the variation of the index in a foam has a pronounced effect on the hardness of the final foam. This increase in hardness has been shown to be directly related to increased covalent cross-linking resulting from more complete consumption of isocyanate reactive sites caused by the presence of excess isocyanate groups. However beside the desired increase of hardness there can be diverse severe disadvantages. Usually an increased index also leads to a bad air permeability of the foam due to the strong network and the hindered cell opening. Increased index also means higher TDI loading, resulting in a raised core temperature of the foam as well as increased foam costs. This higher core temperature might lead to scorch or, in worst case scenarios, spontaneous self-ignition of the foam block. A second method to strengthen the polymeric network is to accelerate the gelling reaction which means using an increased amount of tin catalyst. This results in higher foam costs and may also lead to a tighter foam structure. A third way is the use of cross-linking additives. Cross-linking additives are highly functionalized hydrogen-containing compounds reacting with isocyanates. However according to experience a usage of cross-linking additives also leads to very tight foam structures. Tight foams also show a certain increase in hardness when compared to the same foam grade with open cell structure. Nevertheless for most flexible foam applications tight foam structures are not desired. Besides the bad breathability which is especially undesired for mattresses, tight foams tend to have a declined durability. Closed foam cells will be crushed when the foam is frequently compressed and the so-called “fake” hardness will be lost. Additionally tight foams show a bad resiliency which lowers the comfort of the cushion.

OBJECTIVE

A multitude of applications require the foam to have well controlled mechanical properties. Hardness is especially a quality characteristic in many parts of the world. Since an increase in hardness is not readily possible without major impact on other foam physical properties and various disadvantages arise when applying the common ways to gain hardness of flexible foams, there appears to be a significant demand in the market for strategies to generate permanent hardness of flexible PU foams without tightening the foam. This paper presents a new ORTEGOL[®] product to combine low use level with a significant increase in hardness without major impact on the porosity of the foam and other foam physical properties. Foamers are able to save costs by reducing the amount of polymeric polyol, the tin catalyst or the TDI loading. Due to the potential reduction of TDI borderline formulations may be processed more safely and scorch issues might be reduced.

EXPERIMENTAL

Foams produced for this study were made either by hand pouring in the laboratory or by discontinuous box foam machine trials. In order to evaluate the hardening potency of the new product and its influence on the porosity, Evonik has developed two critical base formulations resulting in foams with different densities (table 2). The final foam density of formulation 1 is 16 kg/m³ (1.0 pcf), and formulation 2 leads to foams with a density of 25 kg/m³ (1.6 pcf). For the base trials, the index of the formulations 1 and 2 and the use level of tin catalyst were kept constant (as indicated in table 2). In further experiments the index and the dosage of KOSMOS[®] 29 were varied in order to analyze the potential savings of TDI or tin catalyst. Foams of formulation 1 were made using 200 g whereas foams of formulation 2 were made using 400 g of polyol with the other constituents of the formulation scaled accordingly. Here, for example, 1.0 part of a component means 1 g of this substance per 100 g of polyol. Water, catalysts and silicone surfactants were added to the polyol and mixed together by stirring at 1000 rpm for 60 s. After addition of the isocyanate, the mixture was stirred at 2500 rpm for 7 seconds. The liquid material was then poured into a paper-lined wooden box (base area: 30 cm x 30 cm). Foams were allowed to cure for 24 hours and cut afterwards. The following measurements were taken and recorded: rise time, height, settling after 3 min, foam density, air permeability of the foam, CLD or ILD hardness at 40% compression after 24 hours, cell size, ball rebound, compression set after 90% compression at 70°C for 22 hours, tensile strength and elongation at break. OH-numbers were determined according to Ph. Eur. 2.5.3 Method A and expressed as mg KOH/g.

| <i>Table 2. Base formulations for conventional flexible slabstock polyether foams. The amounts of raw materials and additives are given in parts per hundred parts (pphp).</i> | | |
|--|--------------------------------|--------------------------------|
| Formulations | Formulation 1 | Formulation 2 |
| Density | 16 kg/m ³ (1.0 pcf) | 25 kg/m ³ (1.6 pcf) |
| Polyol OHN 48 | 100 – X | - |
| Polyol OHN 56 | - | 100 – X |
| Total Water | 5.2 | 3.8 |
| TEGOSTAB® B 8158 | 1.3 | 0.8 |
| TEGOAMIN® 33 | 0.15 | - |
| TEGOAMIN® B 75 | - | 0.15 |
| KOSMOS® 29 | 0.25 | 0.18 |
| Methylene Chloride | 7.5 | - |
| ORTEGOL® HA1 (OHN 193) | X | X |
| TDI Index | <110> | <110> |

The possibility to reduce scorch in borderline formulations was evaluated with formulation 3 (table 3). For that foams produced with formulation 3 were stressed thermally in a microwave oven in order to simulate the thermal stress of production blocks. The index was reduced in comparison to a reference and in order to maintain hardness three parts of the hardening additive were added. Two reference foams were produced. First one with index <105>, the other one with index <110>. Three minutes after pouring the risen foam was placed in the microwave oven and irradiated for 80 s at 1000 W. In the following the foams were cut vertically and discoloration was evaluated by means of visual inspection.

| <i>Table 3. Borderline formulation for the evaluation of scorch of conventional flexible slabstock polyether foams. The amounts of raw materials and additives are given in parts per hundred parts (pphp).</i> | |
|---|--------------------------------|
| Formulations | Formulation 3 |
| Density | 21 kg/m ³ (1.3 pcf) |
| Polyol OHN 48 | 100 – X |
| Total Water | 4.8 |
| TEGOSTAB® B 8232 | 1.0 |
| TEGOAMIN® 33 | 0.20 |
| KOSMOS® 29 | 0.22 |
| TDCPP Flame Retardant | 10 |
| ORTEGOL® HA1 (OHN 193) | X |
| TDI Index | <variable> |

Since one possible application of the hardening additive is the (partial) replacement of polymeric polyol, Formulation 4 was developed (table 4). The capability of the new ORTEGOL® to harden the foam was compared with the foam hardness obtained by using polymeric polyol. Formulation 4 is based on 400 g polyol.

| <i>Table 4. Formulation for polymer polyol containing conventional flexible slabstock polyether foams. The amounts of raw materials and additives are given in parts per hundred parts (pphp).</i> | |
|--|--------------------------------|
| Formulations | Formulation 4 |
| Density | 24 kg/m ³ (1.5 pcf) |
| Polyol OHN 48 | 100 – Y – X |
| Polyol OHN 28, SAN 42% | Y |
| Total Water | 4.0 |
| TEGOSTAB® B 8002 | 1.0 |
| TEGOAMIN® DMEA | 0.25 |
| KOSMOS® 29 | 0.20 |
| ORTEGOL® HA1 (OHN 193) | X |
| TDI Index | <105> |

In order to get an idea of the quality of hardness (real vs. “fake” hardness) and therefore of the durability of the resulting foams we produced some test foams in a discontinuous box foam machine (vol. 1 m³) according to formulation 5 (based on 9 kg of polyol), given in table 5 and performed a fatigue test. This fatigue was realized on the basis of the international standard 3385-1975 (E) [4]. The thickness and hardness measurements obtained before the test are compared to measurements after fatigue. The percentage loss is used to describe the fatigue durability performance. Hardness is measured as ILD 40% hardness before and after 80.000 times of 70% compression.

| Table 5. Base formulation for conventional flexible slabstock polyether foams being used for a fatigue test according to ISO 3385-1975 (E). The amounts of raw materials and additives are given in parts per hundred parts (pphp). | |
|---|--------------------------------|
| Formulations | Formulation 5 |
| Density | 16 kg/m ³ (1.0 pcf) |
| Polyol OHN 56 | 100 – X |
| Total Water | 5 |
| Methylene Chloride | 7.84 |
| TEGOSTAB® B 8228 | 1.68 |
| TEGOAMIN® 33 | 0.13 |
| KOSMOS® 29 | variable |
| ORTEGOL® HA1 (OHN 193) | X |
| TDI Index | <115> |

In all formulations the amount of polyol in pphp is offset against the amount of ORTEGOL® HA1 and the amount of TDI is adjusted. Mechanical analysis (CLD 40% hardness as well as ILD 40% hardness, tensile strength and elongation at break) has been done by using a Zwick 1445 Test Machine. Hardness is either measured as CLD 40% hardness (expressed as *kPa*) or ILD 40% hardness (expressed as *N*) after 24 h. Resiliency was determined by the ball rebound test. Rise measurement was made by using Dr. Wehrhahn Ultrasonic Foam Rise Detection equipment. Cell structure analysis has been made by a flatbed scanner and image analysis software (a4i from Soft Imaging Systems). Porosity measurements were made by using the back pressure method. A constant air stream of 480 l/h is forced to flow through a 5 cm thick sheet of the foam, cut perpendicular to the rise direction. The resulting back pressure is measured in *mm water column*. High values indicate a very tight cell structure. The range is from 1 mm water column (very open) to > 300 mm water column (very tight).

RESULTS AND DISCUSSION

Investigation of the hardening potential of ORTEGOL® HA1 and its influence on the air permeability

| Table 6. Foam physical properties of foams based on formulation 1 containing 3 pphp and 5 pphp of ORTEGOL® HA1 compared to a reference containing no hardening additive. | | | | |
|--|----------------------|------------|------------|------------|
| Entry | | 1 | 2 | 3 |
| | | Reference | 3 pphp HA1 | 5 pphp HA1 |
| Polyol OHN 48 | [pphp] | 100 | 97 | 95 |
| TDI Index | | <110> | <110> | <110> |
| Rise time | [s] | 85 | 99 | 107 |
| Height | [cm] | 24.6 | 24.4 | 24.3 |
| Settling | [cm] | 0.2 | 0.2 | 0.3 |
| Density | [kg/m ³] | 15.9 | 16.0 | 16.0 |
| Porosity | [mm] | 23 | 30 | 74 |
| CLD Hardness 40% (24 h) | [kPa] | 2.5 | 3.1 | 3.4 |
| Increase in Hardness | [%] | - | 25 | 35 |
| Cells | [cm ⁻¹] | 12 | 12 | 12 |
| Ball Rebound | [%] | 30 | 29 | 24 |
| Compression Set (90%/70°C/22 h) | [%] | 3.1 | 6.7 | 7.3 |
| Tensile Strength | [kPa] | 74.6 | 80.3 | 80.0 |
| Elongation at Break | [kPa] | 83.4 | 89.4 | 87.0 |

In order to investigate the hardening potential of ORTEGOL® HA1 and its impact on the porosity of the obtained foams the new foam hardening additive was used in two different formulations, resulting in foams of different densities (formulations 1 and 2, table 2). In a first study the use level of the hardener was chosen to be 3 and 5 pphp and foams were compared to a reference containing no foam hardening additive. The results based on formulation 1 are shown in table 6 and figure 4.

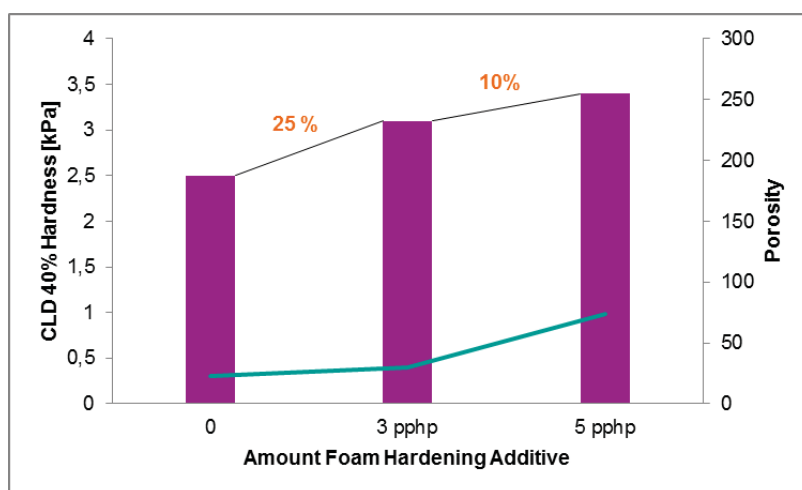


Figure 4. Hardness and porosity of foams based on formulation 1 containing 0, 3 and 5 pphp of ORTEGOL® HA1.

When using three parts of ORTEGOL® HA1 a CLD 40% hardness of 3.1 kPa could be obtained (entry 2, table 6) which corresponds to an increase of 25% compared to the reference (CLD 40 hardness 2.5 kPa, entry 1, table 6). When adding five parts of hardening additive hardness could even be enhanced up to 3.4 kPa (35% increase, entry 3, table 6). With a backpressure porosity of 30 mm the foam containing three parts of hardener is almost as open as the reference (23 mm backpressure H₂O). With five parts of ORTEGOL® HA1 a slight decrease in air permeability to 72 mm H₂O was observed.

Table 7 shows the foam physical properties of foams based on formulation 2. The results are illustrated in figure 5. Again a reference foam without hardening additive was compared to foams containing three and five parts of hardener, respectively. An increase of hardness of 25% was obtained when adding three parts of ORTEGOL® HA1 (3.6 kPa to 4.5 kPa) whereas porosity nearly stayed constant (15 mm H₂O compared to 21 mm H₂O, entries 4 and 5, table 7). A further enhancement of CLD 40% hardness to 4.8 kPa could be observed when adding five parts of hardener. Here again a slightly tighter foam with 52 mm backpressure porosity resulted (entry 6, table 7).

Table 7. Foam physical properties of foams based on formulation 2 containing 3 pphp and 5 pphp of ORTEGOL® HA1 compared to a reference containing no hardening additive.

| Entry | | 4 | 5 | 6 |
|---------------------------------|----------------------|-----------|------------|------------|
| | | Reference | 3 pphp HA1 | 5 pphp HA1 |
| Polyol OHN 56 | [pphp] | 100 | 97 | 95 |
| TDI Index | | <110> | <110> | <110> |
| Rise time | [s] | 107 | 106 | 104 |
| Height | [cm] | 32.4 | 32.7 | 32.7 |
| Settling | [cm] | 0.5 | 0.6 | 0.3 |
| Density | [kg/m ³] | 24.2 | 24.6 | 24.6 |
| Porosity | [mm] | 15 | 21 | 52 |
| CLD Hardness 40% (24 h) | [kPa] | 3.6 | 4.5 | 4.8 |
| Increase in Hardness | [%] | - | 25 | 34 |
| Cells | [cm ⁻¹] | 12 | 12 | 12 |
| Ball Rebound | [%] | 35 | 32 | 21 |
| Compression Set (90%/70°C/22 h) | [%] | 3.8 | 5.5 | 4.5 |
| Tensile Strength | [kPa] | 82.0 | 88.8 | 84.6 |
| Elongation at Break | [kPa] | 88.3 | 80.7 | 69.6 |

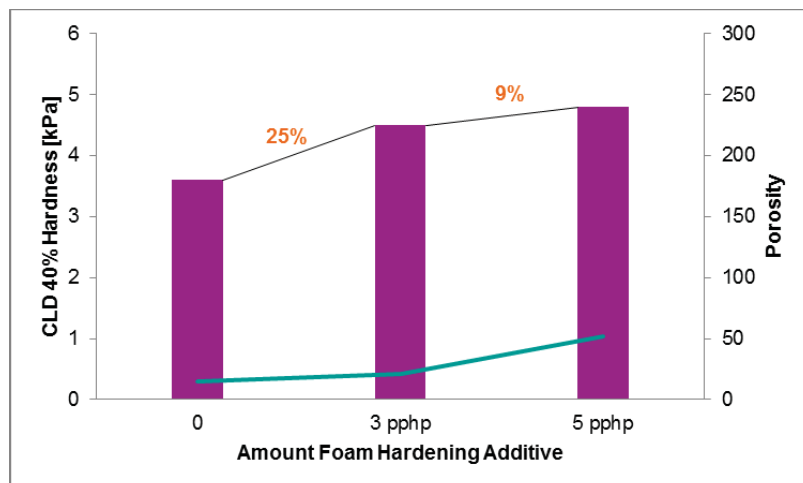


Figure 5. Hardness and porosity of foams based on formulation 2 containing 0, 3 and 5 pphp of ORTEGOL® HA1.

Investigation of the potential savings of TDI and tin catalyst by use of ORTEGOL® HA1

In the following, formulation 1 was used again producing foams based on different indices (thus with different TDI loadings). Foams were prepared using 3 pphp of ORTEGOL® HA1 and compared to reference foams containing no hardening additive. The results are shown in table 8 and figure 6. Starting with <103> we increased the index up to <118>. Obviously an increase of the index led to an increase of hardness of the resulting foams (2.2 kPa to 3.3 kPa for the reference foams without ORTEGOL® HA1 (entry 7 and 19, table 8) and 2.4 kPa to 3.9 kPa in the case of the use of 3 pphp foam hardening additive (entry 8 and 20, table 8)). Also a trend to tighter foam structures was clearly obtained (10 to 49 mm H₂O and 17 to 128 mm H₂O, respectively). We obtained same hardness and very similar porosity for the following foams:

- 1a)** Index <110>, 0 pphp ORTEGOL® HA1 (CLD 40% hardness 2.5 kPa, porosity 25 mm H₂O, entry 13, table 8) and
- 1b)** Index <105>, 3 pphp ORTEGOL® HA1 (CLD 40% hardness 2.5 kPa, porosity 30 mm H₂O, entry 10, table 8)
- 2a)** Index <115>, 0 pphp ORTEGOL® HA1 (CLD 40% hardness 3.1 kPa, porosity 29 mm H₂O, entry 17, table 8) and
- 2b)** Index <110>, 3 pphp ORTEGOL® HA1 (CLD 40% hardness 3.0 kPa, porosity 32 mm H₂O, entry 14, table 8)

For this formulation it was possible to reduce the index by at least 5 points which corresponds to a reduction of the TDI loading of at least 4% when adding three parts of ORTEGOL® HA1 without a major impact on hardness or porosity of the resulting foams.

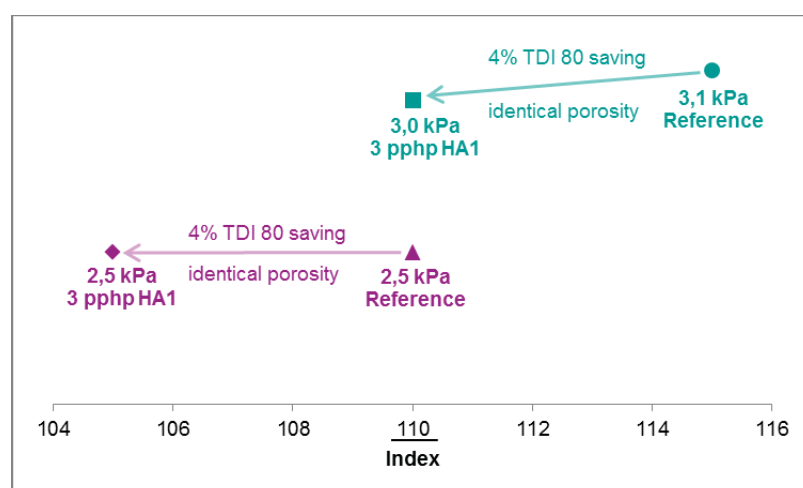


Figure 6. Variation of hardness of foams based on formulation 1 with reduction of the index and addition of 3 pphp of ORTEGOL® HA1.

| Table 8. Foam physical properties of foams based on formulation 1. Reference foams were produced without hardening additive and compared to foams containing 3 pphp of ORTEGOL® HA1. The index was varied from <103> to <118>. | | | | | | | | | | | | | | | |
|--|---------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|
| Entry | | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |
| | | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 |
| Polyol OHN 48 | [pphp] | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 |
| TDI Index | | <103> | | <105> | | <108> | | <110> | | <113> | | <115> | | <118> | |
| KOSMOS® 29 | [pphp] | 0.25 | | 0.25 | | 0.25 | | 0.25 | | 0.25 | | 0.25 | | 0.25 | |
| Rise time | [s] | 96 | 104 | 93 | 101 | 90 | 103 | 89 | 102 | 85 | 100 | 85 | 94 | 84 | 92 |
| Height | [cm] | 22.5 | 22.2 | 23.0 | 23.8 | 23.8 | 24.1 | 23.8 | 23.2 | 24.8 | 23.9 | 25.0 | 24.7 | 26.1 | 26.0 |
| Settling | [cm] | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Density | [kg/m³] | 16.7 | 17.0 | 16.5 | 16.6 | 16.6 | 16.6 | 16.5 | 16.9 | 16.4 | 16.5 | 16.3 | 16.2 | 16.2 | 16.2 |
| Porosity | [mm] | 10 | 17 | 13 | 30 | 16 | 28 | 25 | 32 | 20 | 38 | 29 | 42 | 49 | 128 |
| CLD 40% (24 h) | [kPa] | 2.2 | 2.4 | 2.2 | 2.5 | 2.4 | 2.8 | 2.5 | 3.0 | 2.9 | 3.1 | 3.1 | 3.6 | 3.3 | 3.9 |
| Cells | [cm⁻¹] | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

| Table 9. Foam physical properties of foams based on formulation 1. Reference foams were produced without hardening additive and compared to foams containing 3 pphp of ORTEGOL® HA1. The tin level was varied from 0.20 to 0.30 pphp KOSMOS® 29. | | | | | | | | | | | |
|--|---------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|
| Entry | | 21 | 22 | 23 | 24 | 25 | 26 | 27 | 28 | 29 | 30 |
| | | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 |
| Polyol OHN 48 | [pphp] | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 |
| TDI Index | | <110> | | <110> | | <110> | | <110> | | <110> | |
| KOSMOS® 29 | [pphp] | 0.20 | | 0.22 | | 0.25 | | 0.28 | | 0.30 | |
| Rise time | [s] | 104 | 106 | 98 | 101 | 91 | 96 | 83 | 92 | 83 | 83 |
| Height | [cm] | 22.3 | 22.0 | 23.0 | 23.7 | 22.6 | 23.2 | 23.6 | 23.8 | 23.7 | 24.0 |
| Settling | [cm] | 0.0 | -0.2 | 0.0 | 0.0 | -0.1 | -0.2 | 0.0 | -0.2 | 0.3 | -0.2 |
| Density | [kg/m³] | 16.2 | 16.2 | 16.0 | 16.0 | 16.3 | 15.9 | 15.8 | 16.0 | 15.5 | 15.8 |
| Porosity | [mm] | 12 | 16 | 12 | 22 | 13 | 23 | 16 | 54 | 19 | 60 |
| CLD 40% (24 h) | [kPa] | 2.9 | 3.5 | 3 | 3.8 | 3.2 | 3.9 | 3.2 | 3.9 | 3.3 | 4.1 |
| Cells | [cm⁻¹] | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

Table 9 shows the results when foams based on formulation 1 were prepared with different levels of tin catalyst. Again foams were produced using 3 pphp of ORTEGOL® HA1 and compared to reference foams containing no hardening additive. The loading of KOSMOS® 29 was varied from 0.20 parts to 0.30 parts. The effects on hardness and porosity are also illustrated in figure 7. A loading of tin catalyst of 0.20 parts led to a CLD 40% hardness of 2.9 kPa when no hardening additive was in use (entry 21, table 9) and to a hardness of 3.5 kPa when three parts of hardener were added (entry 22, table 9). In the case of 0.25 parts of KOSMOS® 29 a hardness of 3.2 kPa and 3.9 kPa was obtained, respectively (entries 25 and 26, table 9). With use level of 0.30 parts of tin we observed a hardness of 3.3 kPa and 4.1 kPa, respectively (entries 29 and 30, table 9). Porosity varied from 12 to 19 mm H₂O without hardening additive and from 16 to 60 mm backpressure when three parts of ORTEGOL® HA1 were added. In the graph of figure 7 two foams are compared:

- a) 0.25 pphp KOSMOS® 29, 0 pphp ORTEGOL® HA1 (entry 25, table 9) and
- b) 0.20 parts KOSMOS® 29, 3 pphp ORTEGOL® HA1 (entry 22, table 9).

Even with a reduction of the loading of tin catalyst from 0.25 parts to 0.20 parts (saving of 20% tin) the hardness of the foam could be increased by 10% (3.2 kPa without hardening additive and 3.5 kPa when three parts of ORTEGOL® HA1 were used) without negative impact on the porosity (13 mm backpressure porosity of the reference foam compared to 16 mm H₂O in the case of use of 3 pphp hardener) when three parts of hardening additive was used.

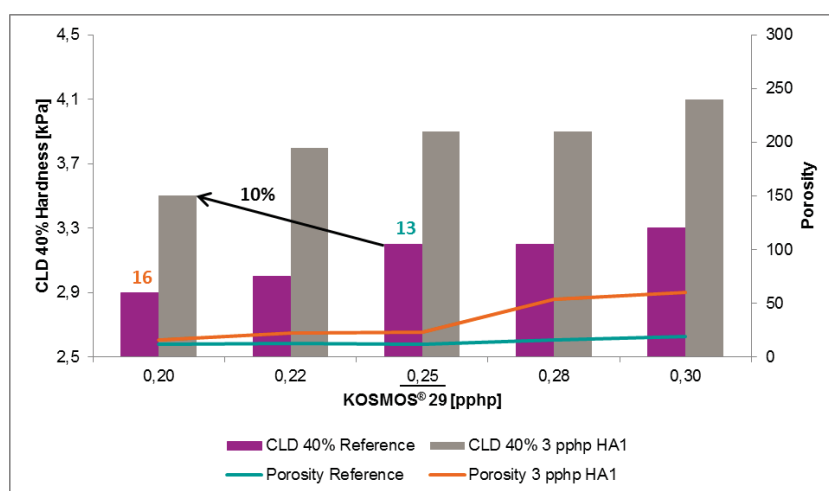


Figure 7. Variation of hardness and porosity of foams based on formulation 1 with reduction of the use level of KOSMOS® 29 and the addition of 3 pphp of hardening additive.

As before also index and tin level were varied in formulation 2. The outcomes are summarized in the tables 10 and 11 and figures 8 and 9. The index was again varied from <103> to <118> whereas the tin level was changed from 0.12 part to 0.23 parts. When varying the index again two couples of foams with similar porosity and hardness were compared:

- 1a) Index <110>, 0 pphp ORTEGOL® HA1 (CLD 40% hardness 3.6 kPa, porosity 13 mm H₂O, entry 37, table 10) and
- 1b) Index <103>, 3 pphp ORTEGOL® HA1 (CLD 40% hardness 3.5 kPa, porosity 21 mm H₂O, entry 32, table 10)
- 2a) Index <118>, 0 pphp ORTEGOL® HA1 (CLD 40% hardness 4.4 kPa, porosity 26 mm H₂O, entry 43, table 10) and
- 2b) Index <110>, 3 pphp ORTEGOL® HA1 (CLD 40% hardness 4.5 kPa, porosity 22 mm H₂O, entry 38, table 10)

For this formulation a reduction of 7 – 8 index points was possible without major changes to porosity and hardness when three parts of the new hardening additive were used. This corresponds to a TDI saving of at least 5%.

| Table 10. Foam physical properties of foams based on formulation 2. Reference foams were produced without hardening additive and compared to foams containing 3 pphp of ORTEGOL® HA1. The index was varied from <103> to <118>. | | | | | | | | | | | | | | | |
|---|---------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|
| Entry | | 31 | 32 | 33 | 34 | 35 | 36 | 37 | 38 | 39 | 40 | 41 | 42 | 43 | 44 |
| | | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 |
| Polyol OHN 56 | [pphp] | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 |
| TDI Index | | <103> | | <105> | | <108> | | <110> | | <113> | | <115> | | <118> | |
| KOSMOS® 29 | [pphp] | 0.18 | | 0.18 | | 0.18 | | 0.18 | | 0.18 | | 0.18 | | 0.18 | |
| Rise time | [s] | 117 | 117 | 116 | 114 | 112 | 110 | 113 | 110 | 104 | 104 | 104 | 104 | 100 | 101 |
| Height | [cm] | 30.5 | 31.1 | 31.4 | 31.5 | 32.6 | 32.5 | 32.0 | 32.7 | 33.2 | 33.8 | 33.5 | 33.6 | 34.5 | 34.9 |
| Settling | [cm] | 0.7 | 0.4 | 0.6 | 0.5 | 0.6 | 0.4 | 0.6 | 0.3 | 0.6 | 0.2 | 0.7 | 0.3 | 0.6 | 0.2 |
| Density | [kg/m³] | 24.4 | 24.5 | 24.4 | 24.7 | 24.2 | 24.4 | 24.4 | 24.8 | 24.9 | 24.2 | 23.8 | 24.2 | 23.8 | 24.0 |
| Porosity | [mm] | 13 | 21 | 14 | 29 | 13 | 22 | 13 | 22 | 26 | 49 | 20 | 74 | 26 | 117 |
| CLD 40% (24 h) | [kPa] | 3 | 3.5 | 3.3 | 3.8 | 3.5 | 4.3 | 3.6 | 4.5 | 3.8 | 5.1 | 3.8 | 4.8 | 4.4 | 5.8 |
| Cells | [cm⁻¹] | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

| Table 11. Foam physical properties of foams based on formulation 2. Reference foams were produced without hardening additive and compared to foams containing 3 pphp of ORTEGOL® HA1. The tin level was varied from 0.12 to 0.23 pphp KOSMOS® 29. | | | | | | | | | | | |
|---|---------|-------|------------|-------|------------|-------|------------|-------|------------|-------|------------|
| Entry | | 45 | 46 | 47 | 48 | 49 | 50 | 51 | 52 | 53 | 54 |
| | | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 |
| Polyol OHN 56 | [pphp] | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 | 100 | 97 |
| TDI Index | | <110> | | <110> | | <110> | | <110> | | <110> | |
| KOSMOS® 29 | [pphp] | 0.12 | | 0.15 | | 0.18 | | 0.20 | | 0.23 | |
| Rise time | [s] | 140 | 141 | 124 | 122 | 113 | 112 | 105 | 104 | 96 | 97 |
| Height | [cm] | 30.3 | 30.7 | 31.6 | 32.0 | 31.4 | 31.6 | 32.6 | 33.2 | 33.2 | 33.7 |
| Settling | [cm] | 0.2 | 0.7 | 0.6 | 0.7 | 0.5 | 0.4 | 0.6 | 0.2 | 0.3 | 0.0 |
| Density | [kg/m³] | 25.1 | 25.0 | 24.4 | 24.6 | 24.6 | 24.9 | 24.1 | 24.2 | 24.3 | 24.1 |
| Porosity | [mm] | 13 | 19 | 15 | 21 | 15 | 22 | 19 | 48 | 37 | 230 |
| CLD 40% (24 h) | [kPa] | 3.3 | 3.9 | 3.4 | 4.3 | 3.7 | 4.6 | 3.8 | 4.8 | 3.9 | 5.5 |
| Cells | [cm⁻¹] | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

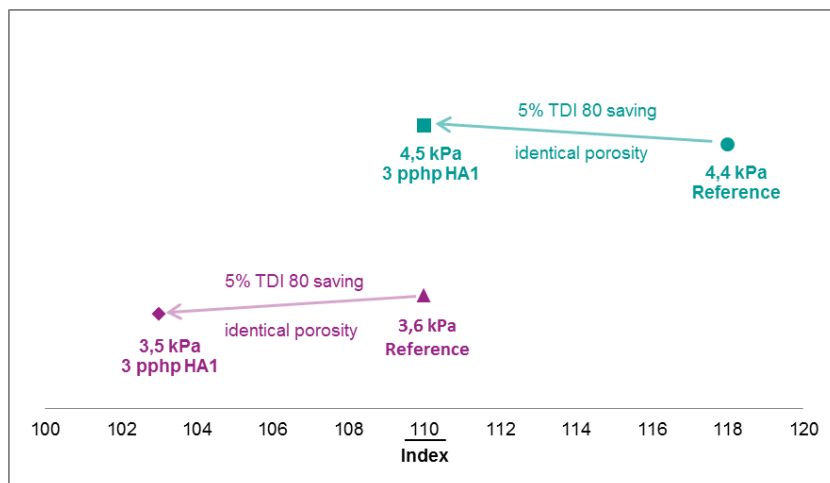


Figure 8. Variation of hardness of foams based on formulation 2 with reduction of the index and addition of 3 pphp of ORTEGOL® HA1.

When the tin level of formulation 2 was varied (constant index of <110>) from 0.12 pphp to 0.23 pphp a CLD 40% hardness of 3.3 kPa to 3.9 kPa was obtained when no hardening additive was used (entries 45 and 53, table 11) whereas hardness could be increased to 3.9 kPa to 5.5 kPa in the case of hardener usage (entries 46 and 54, table 11). Again two foams with similar porosity were compared in the graph of figure 9:

- a) 0.18 pphp KOSMOS® 29, 0 pphp ORTEGOL® HA1 (entry 49, table 11) and
- b) 0.12 pphp KOSMOS® 29, 3 pphp ORTEGOL® HA1 (entry 46, table 11).

With the reduction of tin catalyst from 0.18 pphp to 0.12 pphp (saving of 33% tin) and the addition of three parts of ORTEGOL® HA1 even a slightly harder foam (+ 5% hardness, 3.7 kPa CLD 40% hardness) was obtained compared to the reference foam without hardening additive (3.9 kPa CLD 40% hardness). Additionally only little impact on the porosity was observed (reference 15 mm H₂O compared to 22 mm H₂O in the case of hardener usage).

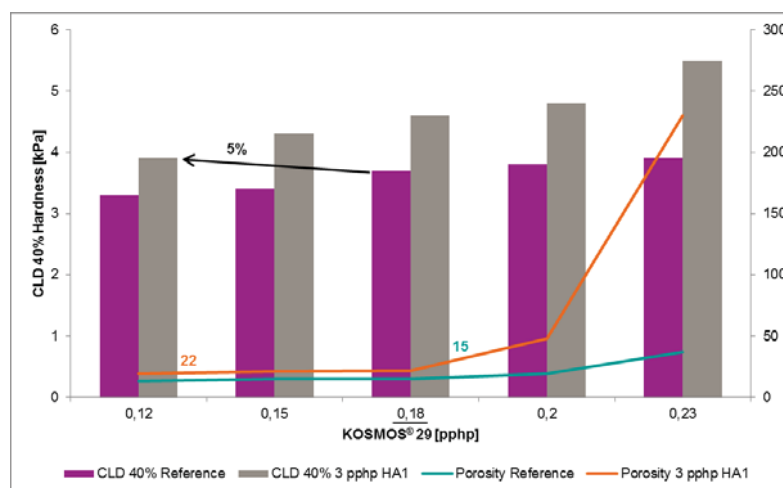


Figure 9. Variation of hardness and porosity of foams based on formulation 2 with reduction of the use level of KOSMOS® 29 and the addition of 3 pphp of hardening additive.

Reducing of scorch in borderline formulations by usage of ORTEGOL® HA1 and reducing the TDI loading

As already mentioned above it is possible to save TDI by the usage of ORTEGOL® HA1. One application of that could be the reduction of the core temperature of foam blocks in order to reduce scorch or to minimize the risk of self-ignition. For that the possibility to reduce scorch in borderline formulations was evaluated with formulation 3. Two pairs of foams were prepared

with reference foams of index <105> and <110>, respectively. Afterwards the index was reduced in comparison to the reference and in order to maintain hardness three parts of the hardening additive were added. Foaming results are shown in table 12. The first pair of foams (entry 55, table 12, index <105> & entry 56, table 12, index <100>, 3 pphp HA1) showed nearly identical physical properties. Very similar values for CLD 40% hardness (3.6 kPa vs. 3.5 kPa) and air permeability (9 mm H₂O vs. 12 mm H₂O) were obtained. The same trend was obtained with regard to the second couple of foams (entry 57, table 12, index <110> & entry 58, table 12, index <105>, 3 pphp HA1). Again very similar hardness (4.0 kPa vs. 4.1 kPa) and porosity (9 mm H₂O vs. 12 mm H₂O) of the resulting foams were obtained.

| Table 12. Foam physical properties of two couples of foams based on formulation 3 containing 3 pphp ORTEGOL® HA1 compared to a reference with increased index containing no hardening additive. | | | | | |
|---|---------------------|-----------|------------|-----------|------------|
| Entry | | 55 | 56 | 57 | 58 |
| | | Reference | 3 pphp HA1 | Reference | 3 pphp HA1 |
| Polyol OHN 48 | [pphp] | 100 | 97 | 100 | 97 |
| TDI Index | | <105> | <100> | <110> | <105> |
| Rise time | [s] | 84 | 88 | 80 | 85 |
| Height | [cm] | 22.7 | 22.4 | 22.3 | 23.2 |
| Settling | [cm] | 0.3 | 0.2 | 0.2 | 0.2 |
| Porosity | [mm] | 9 | 12 | 9 | 12 |
| CLD Hardness 40% (24 h) | [kPa] | 3.6 | 3.5 | 4.0 | 4.1 |
| Cells | [cm ⁻¹] | 12 | 12 | 12 | 12 |

In the following the risen foams were placed in the microwave oven and stressed thermally (irradiation for 80 s at 1000 W). The foams were cut vertically and discoloration was evaluated by means of visual inspection (figure 10). For both couples of foams a significant reduction of scorch and therefore a significant reduction of the core temperature of the foam was observed when the index was reduced by 5 points whereas only negligible effects on the foam physical properties were obtained.



Figure 10. Scorching test of two pairs of foams made according to formulation 3. Reduction of the index and an addition of ORTEGOL® HA1 to maintain hardness led to a significant reduction of scorch (microwave irradiation: 80 s, 1000 W).

Investigation of the potential savings of polymeric polyol by use of ORTEGOL® HA1

Hard foams often are obtained by using polymer solids containing polyol. In order to save costs it is often desired to reduce the amount of such polymeric polyol in hard foam formulations. In the following, formulation 4 was used to compare the hardening potential of the new ORTEGOL® to the hardness increase as a result of the usage of polymeric polyol. For that a polymeric polyol with an OH number of 28 mg KOH/g and a polymer solid content (SAN) of 42% was chosen. Five pairs of foams were prepared, in each case one without and one with three parts of hardening additive: Firstly two foams were made containing no polymeric polyol but only conventional polyol. Without the use of hardening additive a CLD 40% hardness of 3.8 kPa was obtained (entry 59, table 13) and by using three parts of the new ORTEGOL® an increase of hardness to 4.8 kPa could be achieved (entry 60, table 13). Next two foams which consisted of 100% polymeric polyol were produced (entry 61 and 62, table 13). Logically very hard foams were generated. Without hardening additive a hardness of 10.5 kPa whereas with three parts of ORTEGOL® HA1 a hardness of 12.3 kPa could be obtained. In the following three pairs of foams were made out of a mixture of conventional and polymeric polyol, ratios were 50 : 50, 70 : 30 and 90 : 10. In the cases where ORTEGOL® HA1 was in use, the amount of *conventional* polyol in pphp was offset against the amount of foam hardening additive. Foams consisting of 50% of conventional and 50% of polymeric polyol showed a hardness of 6.6 kPa and 7.9 kPa, respectively (entry 63 and 64, table 13). Foams containing 70% of conventional and 30% of SAN polyol showed an interesting behavior: Without

hardening additive a CLD 40% hardness of 4.8 kPa was obtained (entry 65, table 13), just as much as the hardness of the foam without polymeric polyol containing three parts of ORTEGOL® HA1 (entry 60, table 13). Accordingly it was possible to replace 30 pphp of SAN polyol by three parts of the new hardening additive ORTEGOL® HA1 in this formulation. By adding 3 pphp of hardener to that 30% polymeric polyol containing foam formulation hardness even could be increased to 5.6 kPa (entry 66, table 13). Another remarkable situation is observed when foams were made with 90% of conventional and 10% of SAN polyol: Without hardening additive nearly no increase in hardness was noted (3.9 kPa, entry 67, table 13) compared to the foam containing no polymeric polyol and no foam hardener (3.8 kPa, entry 59, table 13). The same could be seen when three parts of ORTEGOL® HA1 were added. A foam with a CLD 40% hardness of 4.8 kPa was obtained (entry 68, table 13), just the identical hardness as in the case of a foam without SAN polyol containing 3 pphp hardener (entry 60, table 13). Therefore with respect to CLD 40% hardness no benefit in using 10 pphp of polymeric polyol was observed in this formulation. In all cases the resulting foams had a very open cell structure. Air permeability varied from 1 mm H₂O (entry 61, table 13) to 15 mm backpressure (entry 68, table 13).

Table 13. Comparison of the hardening potential of polymeric polyol and ORTEGOL® HA1 according to formulation 4.

| Entry | | 59 | 60 | 61 | 62 | 63 | 64 | 65 | 66 | 67 | 68 |
|------------------------|----------------------|------|---------------|------|---------------|------|---------------|------------|---------------|------|---------------|
| | | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 | Ref. | 3 pphp HA1 |
| Polyol OHN 48 | [pphp] | 100 | 97 | 0 | 0 | 50 | 47 | 70 | 67 | 90 | 87 |
| Polyol OHN 28, SAN 42% | [pphp] | 0 | 0 | 100 | 97 | 50 | 50 | 30 | 30 | 10 | 10 |
| ORTEGOL® HA1 | [pphp] | 0 | 3 | 0 | 3 | 0 | 3 | 0 | 3 | 0 | 3 |
| Rise time | [s] | 93 | 98 | 76 | 78 | 77 | 80 | 83 | 87 | 89 | 91 |
| Height | [cm] | 28.2 | 28.6 | 28.2 | 28.2 | 28.5 | 29.4 | 28.3 | 29.4 | 28.8 | 28.4 |
| Settling | [cm] | 1.1 | 0.9 | 0.9 | 0.5 | 0.5 | 0.6 | 0.7 | 0.7 | 0.9 | 1.0 |
| Density | [kg/m ³] | 25.5 | 26.0 | 25.3 | 25.0 | 25.4 | 25.3 | 25.5 | 25.4 | 25.3 | 25.3 |
| Porosity | [mm] | 9 | 12 | 1 | 4 | 1 | 9 | 4 | 4 | 6 | 15 |
| CLD 40% (24 h) | [kPa] | 3.8 | 4.8 | 10.5 | 12.3 | 6.6 | 7.9 | 4.8 | 5.6 | 3.9 | 4.8 |
| Cells | [cm ⁻¹] | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 | 12 |

Investigation of the durability of foams being prepared by the use of ORTEGOL® HA1

Generally, tight cell structure leads so some hardness increase as well. This hardness however is not a stable hardness when the foam is permanently loaded. In order to differentiate the so-called “fake” hardness from the real long-lasting hardness which is obtained with ORTEGOL® HA1 and thus to evaluate the quality of hardness, we prepared two foams with equivalent porosity and hardness based on formulation 5. In order to be able to work with the same index and to obtain a similar hardness the tin catalyst level had to be slightly adjusted. Both foams were produced at an index of <115>, the reference containing no ORTEGOL® HA1 was made using 0.28 pphp KOSMOS® 29 whereas the foam containing three parts of hardening additive was made using 0.22 pphp KOSMOS® 29. In the following a fatigue test was performed on the basis of ISO 3385-1975 (E). In the test, a 380 mm x 380 mm x 50 mm foam sample was repeatedly intended to 70% of its original thickness at a rate of 70 cycles per minute for 80,000 cycles. The thickness and hardness measurements obtained before and after fatigue were recorded and expressed as *percentage loss of thickness and hardness*, respectively.

Table 14. Fatigue test of two foams based o formulation 5, comparing a reference to one foam containing 3 pphp ORTEGOL® HA1

| Entry | | 69 | 70 |
|-------------------------|--------|-------------|-------------|
| | | Reference | 3 pphp HA1 |
| Polyol OHN 56 | [pphp] | 100 | 97 |
| KOSMOS® 29 | [pphp] | 0.28 | 0.22 |
| Porosity | [mm] | 49 | 43 |
| ILD Hardness 40% (24 h) | [N] | 110 | 113 |
| Fatigue (Thickness) | [%] | 2.0 | 2.0 |
| Fatigue (ILD 40%) | [%] | 24.6 | 16.2 |

As shown in table 14 there was no significant loss of thickness observed when performing the fatigue test. However a major impact could be seen in the change of ILD 40% hardness. When using three parts of hardening additive the loss in ILD 40% hardness could be reduced from 24.6% (entry 69, table 14) to 16.2% (entry 70, table 14). This is a very interesting outcome as many countries have regulations stating a maximum hardness loss at a specific density.

SUMMARY

Many applications require flexible polyurethane foam to have well controlled mechanical properties. Hardness was identified to be one of the properties which are crucial for a multitude of applications. Whereas the reduction of hardness and obtaining a softening effect usually has only limited impact on other foam physical properties, the increase of hardness is often not readily possible without undesirable changes of other foam physical properties. Different ways to increase hardness were discussed, however most of them showing severe disadvantages as for instance an unfavorable influence on the porosity, higher foam costs, an undesirable impact on the emanations of volatile organic compounds (VOCs) or a non-lasting foam hardness which leads to a bad durability of the resulting foam. The new versatile hardening additive ORTEGOL® HA1 was developed in order to meet the requirements for a broad range of different PU foam grades. ORTEGOL® HA1 combines low use level with a significant increase in hardness with minor impact on other foam physical properties. It was possible to increase the hardness of foams of different densities up to 35% whereas porosity nearly stayed constant. In terms of cost saving it was possible to reduce the TDI loading up to 10% and the tin catalyst amount up to more than 30% when using three parts of the new ORTEGOL® additive. A replacement of 30 parts of SAN polymeric polyol was achieved by the use of three parts of hardening additive without changes in other physical properties. This might lead to a significant reduction of costs when high priced polymeric polyol can be reduced. It was further possible to minimize scorch in a borderline formulation by decreasing the TDI index and adjusting hardness by usage of ORTEGOL® HA1. The high quality hardness which can be obtained by using ORTEGOL® HA1 could be proved with the little loss in ILD 40% hardness after a fatigue test performed according to the international standard 3385-1975 (E). These exceptional properties make the new ORTEGOL® Hardening Additive an ideal candidate for the production of hard flexible slabstock foams.

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BIOGRAPHIES

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Dr. Michael Krebs studied chemistry at the University of Stuttgart, Germany and the University Louis Pasteur of Strasbourg, France in a Franco-German double degree course. After completing his studies in France he received his PhD in organic chemistry in the field of natural product synthesis in 2013. In April 2013 he joined the application technology team of the business line Comfort and Insulation of Evonik Industries as a Technical Manager for Flexible Foam. Among others his tasks include the development of new foam stabilizers and performance additives. He is in charge of the quality control of Flexible Slabstock Polyurethane Stabilizers and he is technically responsible for Flexible Polyurethane Additives in Africa.

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Dr. Roland Hubel, born in 1972, studied Chemistry at the Ludwig-Maximilians-University of Munich. He received his PhD in Coordination- and Metal organic Chemistry. After working for one year in the field of genetic engineering as civil servant he joined Degussa Goldschmidt Polyurethane Additives as a research scientist in 2000. After that he was responsible for R&D of polyethers and development and application technology of additives for microcellular foams. Today he is globally responsible for development and application technology for Flexible Slabstock Polyurethane Additives.

Jens Sassenhagen



Jens Sassenhagen started his education as a chemical technician in 1991. From 1994 - 2011 he worked in the production plant for silicone stabilizers at the Th. Goldschmidt AG (later Degussa/Evonik Industries AG). In 2011 he moved to the application technology department for flexible foam additives as a lab technician and was promoted to a Technical Service Manager afterwards. Today he is responsible for technical service for Flexible Slabstock Polyurethane Additives in South East Asia and Africa.