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Development of inks suitable for the manufacturing of micro-scale polyurethane foams

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Abstract

We herein present the development of inkjet inks that have the capability of producing micro-scale polyurethane foams. Inkjet technology can be used as an additive manufacturing tool using small amounts of liquids to form a desired structure. Furthermore, in situ chemical reactions can be carried out using this particular printing technique.

In order to achieve suitable printing inks, viscosity properties were taken as the key limiting factor choosing the ink components. Poly(ethylene glycol) 200 was selected as the main polyol for the reactive inks. Glycerol ethoxylate, also known as star-PEG, was selected as the crosslinking agent due to its three primary hydroxyl groups as well as its good compatibility with PEG200. Variation in star-PEG content was investigated by means of rheometric and tensiometric measurements. For the reactive isocyanate compound, 1,6-hexamethylene diisocyanate (HDI) was chosen due to its high reactivity and low viscosity.

Polyurethane foams (PUF) were prepared in bulk to validate the foam recipe. Small droplets of the inks were then tested by manually placing two drops on top of each other. It was shown that in the absence of mechanical mixing the gelling and blowing reaction still take place. Furthermore, catalytic influences on the reactions were investigated using a central composite experimental design combined with FTIR-ATR spectroscopy. In order to evaluate the spectra, a deconvolution of the Amid I and Amid II area was conducted. The results were evaluated using an analysis of variance to gain models.

Introduction

Processes that are dominated by interfacial interactions often require large surface areas. Porous polymers, a subgroup of porous materials provide the advantage of easy manufacturing and handling compared to crosslinked particle layers. Porous polymers, more specifically polyurethane based foams (PUF), are well known to the industry, having application in the fields of insulation, cushioning or general lightweight materials [1]. PUF also specialize in demanding fields such as adsorber materials [2], scaffolds in tissue engineering [3], [4] or as 3D-carriers for cells [5].

In many of the above mentioned applications spatially resolved structures are of high interest. Yet, most porous materials are manufactured in bulk processes only allowing uniformly distributed properties of the foam.

Additive manufacturing methods would allow assembly of spatially resolved porous polymers leading towards more individualized materials. Recently, reactive inkjet printing [6] has been identified to be suitable for the manufacturing of two and three-dimensional polyurethane structures [7]–[10].

In particular, Kroeber *et al.* witnessed the generation of CO_2 while conducting reactive inkjet printing of an aromatic isocyanate combined with poly(ethylene glycol) 400 [7]. However, the observation was not of interest for their particular study. Therefore, our approach starts at this point, developing inkjet inks that are suitable for the manufacturing of porous structures using a reactive inkjet printing approach.

In order to understand the reactive behavior and catalytic influences of the possible ink formulations, FTIR studies were found to be applicable [7], [11]–[13]. In order to describe the influences of the catalysts to the reaction behavior of the formulations, an experimental design (DoE) was selected to obtain describing models.

Materials and Methods

Bulk preparation of foams

Bulk polyurethane foams were prepared by mixing the correspondent polyol component with the isocyanate component. The polyol mixtures consists of poly(ethylene glycol) 200 (M = 200 g/mol, Merck, Germany), the crosslinking agent glycerol ethoxylate further on referred to as star-PEG (M = 1000 g/mol, Sigma-Aldrich) and a foam stabilizer Xiameter® OFX-0193 Fluid (Dow Corning). As catalysts iron(III)-chloride hexahydrate (p.A., Sigma-Aldrich), 1,4-diazabicyclo[2.2.2]octane (Sigma-Aldrich) and DABCO® BL-11 (Air Products) were used. The isocyanate compound Desmodur® H 1,6-hexamethylene diisocyanate HDI (Covestro, Germany), was purchased. Deionized water was used as the blowing agent. All materials were used without further purification. In order to ensure thorough mixing, the highspeed mixer Dispermat® LC30 (VMA-Getzmann, Germany) together with a dissolver disc (d = 28 mm) was used.

Table 1: Formulation recipe of the prepared polyurethane foam. Isocyanate index 110 water blown foam.

Polyol component	weight (g)
Poly(ethylene glycol) 200	92
Glycerol ethoxylate	8
Deionized Water	2
Iron(III)-chloride hexahydrate	0.67
1,4-Diazabicyclo[2.2.2]octane	0.4
DABCO® BL-11	0.4
Xiameter® OFX-0193 Fluid	2
Total	105.47
Desmodur® H	107.89

The polyol components were weighed in and mixed with a magnetic stirrer until no solid catalyst residue was evident. The

isocyanate compound was then poured into the polyol component and immediate mixing at the Dispermat® was carried out for 60 seconds at 1500 rpm. The mixed foam formulation was placed in a fume hood until the foaming reaction was completed. Table 1 shows the weight fractions of a typical foam formulation.

Ink viscosity and surface tension

The inks viscosity was determined by rotary concentric cylinder rheometry using a Physica Modular Compact MCR301 Rheometer (Anton Paar, Germany) at shear rates from 1 s^{-1} to 1500 s^{-1} at 20 °C. The temperature dependent graphs were recorded at a constant shear rate of 100 s^{-1} using a heat rate of 1.92 K/min. The temperature ranged from 20 °C up to 72 °C.

The inks dynamic surface tension was determined using a bubble pressure tensiometer BP50 (Krüss, Germany). The surface age was varied from 15 ms to 3000 ms at a temperature of 60 °C ± 2 °C. The samples were heated in a controlled water bath. The average of three measurements was taken for each sample.

IR-Spectroscopy

IR characterization was performed on a Vertex 70 FT-IR spectrometer (Bruker, Germany) in combination with a Diamond ATR Unit (DuraSamplIR II, Sens IR Technologies, UK). The samples were recorded from 700 cm^{-1} to 4000 cm^{-1} with a resolution of 4 cm^{-1}. Background measurements were taken with 60 scans while performing the sample measurements, 40 scans were found to be sufficient leading to an acquisition time of one minute per spectra. Each sample was recorded for a total of 45 minutes. In the order of 1 g the isocyanate compound (HDI) was weighed out and added to the corresponding amount of polyol mixture followed by ten seconds of mixing with a vortex-mixer (Lab dancer, IKA, Germany). Four drops of the mixed foam formulation were then transferred to the ATR unit. In order to prevent the liquid from spreading the ATR unit's surface, a small glass cylinder restricted the liquid forming a small reaction vessel on top of the diamond. To help the band assignment, a reaction of HDI and 1,6-hexamethylene diamine (HDA) (Sigma-Aldrich), with an isocyanate to amine ratio of 1, was performed. An ATR spectrum of the formed polyurea powder was recorded one hour after the reaction.

The software Design Expert (Stat-Ease inc., USA) was used to generate a central-composite experimental design (DoE) resulting in 19 experiments varying the concentrations of the three different catalysts within the ink formulations (see Table 2).

Table 2: Variation of the concentration (parts per hundred polyol) forming the experimental space for the DoE.

Concentration (php)	1	2	3	4	5
Iron(III)-chloride hexahydrate	0	0.21	0.67	1.12	1.33
1,4-Diazabicyclo[2.2.2]octane	0	0.13	0.4	0.67	0.8
BL-11	0	0.13	0.4	0.67	0.8

An analysis of variance (ANOVA) was performed to minimize the error of the model and to identify the main effects. The deconvolution of the spectra was carried out with OPUS software (Bruker, Germany) using a Levenberg-Marquardt algorithm. The peak form was selected to be Gaussian and the band width could not exceed 60 cm^{-1}. The selected spectra were baseline corrected using the rubber band method with a maximum of 15 iterations. The threshold for the root mean square error a value of 0.03 was found to be suitable.

Manual drop deposition

In order to place small droplets of the inks on top of each other a pipet with a total volume of 10 µL (Eppendorf, Germany) was used. Additionally, glass slides with dimensions of 76 mm x 26 mm (Thermo Scientific, Germany) were silanized to ensure a hydrophobic surface on which the droplets do not spread. Modification by silanization was carried out as follows. First, the glass slides were rinsed with acetone, isopropanol and followed by deionized water. The dried slides were then activated for 45 minutes at a temperature of 70 °C in a solution of ammonia hydroxide (w = 0.24) (Applichem, Germany) and hydrogen peroxide (w = 0.30) (Applichem, Germany). The mixture contained two parts of the hydrogen peroxide and three parts (w/w) of the ammonia solution. After activation the slides were cleaned in deionized water and dried under a nitrogen stream. Silanization was then performed in a mixture of 50 µL dodecyltrichlorsilane (w = 0.96) (ABCR, Germany) and 100 mL of toluene (ACS grade, J.T. Baker, Germany). The slides were immersed for one hour, rinsed with fresh toluene and dried under a nitrogen stream. Contact angle measurements were carried out using an OCA 25 contact angle measurement device (Data Physics, Germany) at 23 °C.

Results and Discussion

Bulk preparation of foams

In order to confirm the general ability of the reactive inks to form a foamed polyurethane material, a bulk preparation of foams was conducted. Figure 1 shows a foam (see Table 1 for recipe) prepared in bulk.

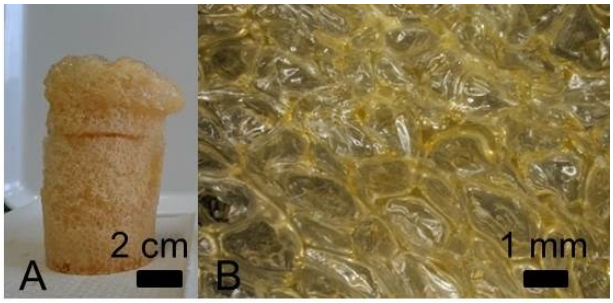

Figure 1: *In bulk prepared foam of the reactive inks. (A) PUF 24 hours after the foaming reaction. (B) Microscopic picture of a cross-section of the foam. The yellowish color is caused by the iron-based catalyst.*

As one can see in part A of Figure 1, a relatively homogenous foaming reaction is evident. The foam has completely filled its vessel and the foam structure is reasonably stable. The foaming roughly starts four minutes after mixing. In part B, a microscopic top view of a cross-section of the obtained foam after the reaction is displayed. Large pores that mainly seem closed are visible. The formulation shows the capability of forming porous polyurethane foams.

Ink viscosity

In order to determine the inks flow behavior and temperature dependent viscosity, rheometric measurements were carried out. Figure 2 shows the flow curves of different contents of the trifunctional star-PEG in order to determine the influence

on the flow behavior of the inks. As all measurements show straight lines, the viscosity has no dependence upon the shear rate in the displayed range. Varying the content of the trifunctional star-PEG up to 20 w% within the formulation results in an increase in viscosity but Newtonian flow behavior is not altered.

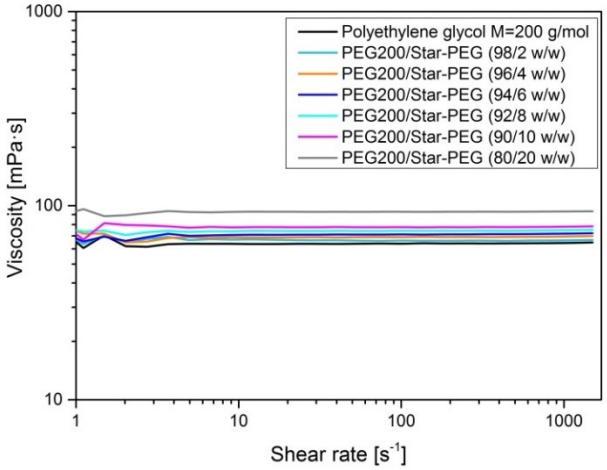

Figure 2: *Double logarithmic flow curves of ink formulations. The content of the trifunctional star-PEG was varied from 2 w% to 20 w%. Newtonian flow behavior is evident at all contents for shear rates up to 1500 s^{-1}.*

However, at room temperature the inks show total viscosities that are higher than the threshold of 20 mPa*s that is necessary for drop formation in most inkjet printers [14].

Therefore, the temperature dependent flow curves were recorded. Figure 3 shows the temperature dependent viscosities of the ink formulations at different contents of star-PEG. As expected, the rise in viscosity with increasing content of star-PEG is still visible. By heating the solutions to a maximum of 72 °C the viscosity falls below the threshold of 20 mPa*s. Depending on the content of star-PEG, a temperature selected between 50 °C and 72 °C will give suitable viscosities for drop formation.

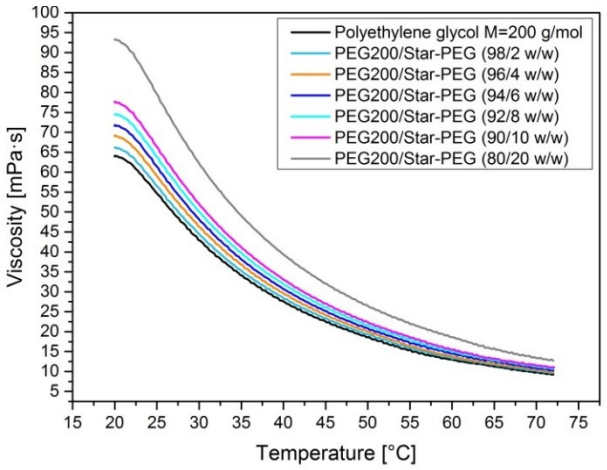

Figure 3: *Temperature dependent viscosity between 20 °C and 72 °C. The content of the trifunctional star-PEG was varied from 2 w% to 20 w%.*

Ink dynamic surface tension

The inks surface tension is important as it influences the drop formation and jetabillity of the ink. As shown in Figure 4 the average surface tension recorded against the surface age of the formed gas bubble is depicted.

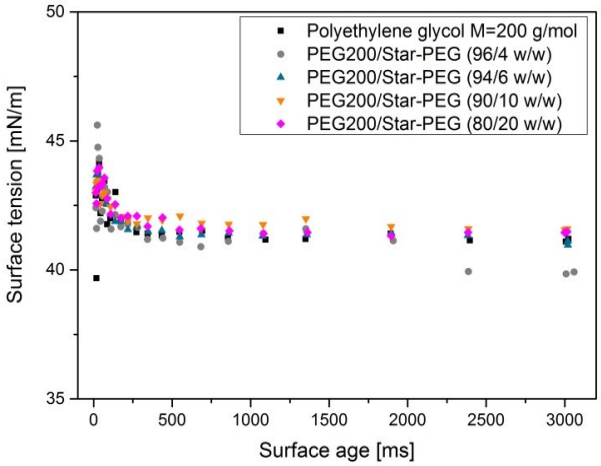

Figure 4: *Surface tension in dependence of the surface age. The content of the trifunctional star-PEG was varied from 4 w% to 20 w%. Surface age ranged from 15 ms to 3000 ms at a temperature of 60 °C ± 2 °C*

Each sample shows slightly dynamic surface tension values between surface ages of 15 ms and 100 ms. Yet, the values range mainly between 45 mN/m and 40 mN/m. A dependence on the content of the trifunctional star-PEG component cannot be observed. Combining the viscosity and surface tension values with the printability model taken from Derby [14], jetabillity of the fluids is possible.

IR-Spectroscopy

The ink formulations contain a mixture of three different catalysts that catalyze parallel reactions occurring during the formation of the foam. Known to catalyze the reaction between the isocyanate and the polyol components iron(III)-chloride as well as diazabicyclooctane (DABCO) were selected [15], [16]. The tertiary amine bis(2-dimethylaminoethyl) ether (Bl-11) was selected as a blowing catalyst, influencing the water and isocyanate reaction.

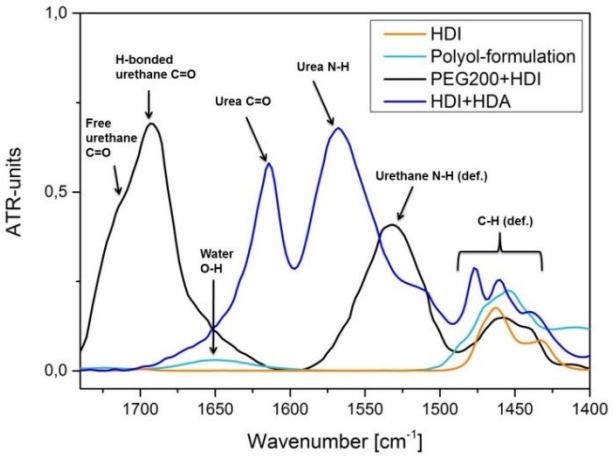

Figure 5: *ATR spectroscopic records of the starting materials HDI and the polyol formulation. For band assignment spectra of pure PEG200 reacted with HDI as well as the urea material formed by the reaction of HDI and its corresponding amine HDA is also displayed.*

Figure 5 shows the band assignments of the starting materials and the formed polyurethane (PEG200+HDI) as well as polyurea (HDI+HDA). In the displayed frequencies HDI

only shows CH-deformation from 1500 cm^{-1} to 1400 cm^{-1}. Additionally, the pure polyol formulation shows a broad water band at 1650 cm^{-1}. Two polyurethane bands are visible at 1720 cm^{-1} (free) and 1695 cm^{-1} (H-bonded).

The reaction of HDI and HDA forms a polyurea that shows a C=O band at 1614cm^{-1} as well as the N-H deformation vibration at 1568 cm^{-1}.

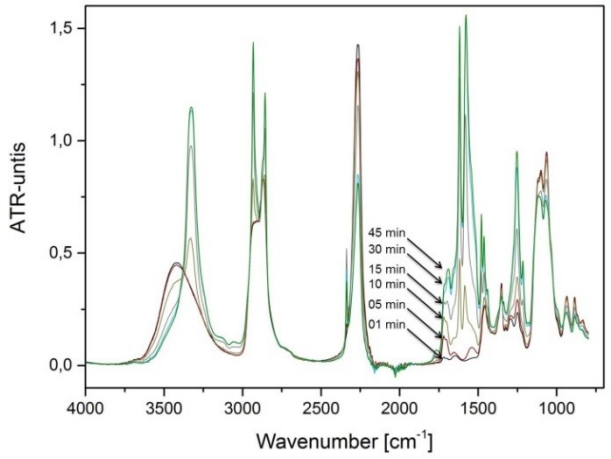

Figure 6: *ATR spectra of in dependence of reaction time. 1, 5, 10, 16, 30 and 45 minutes of reaction time are displayed. The reaction progress can be followed by several changes in band absorptions.*

As one can see in Figure 6, the IR-spectra change significantly as the reaction progresses. Unfortunately, no band was present that was not subject to intensity change during the reaction. Therefore, it was not possible to evaluate the isocyanate band (2267 cm^{-1}) normalized to an internal reference. The carbonyl region (1700 cm^{-1} to 1500 cm^{-1}) was chosen instead. In order to evaluate the overlapping signals, a deconvolution of the spectra was carried out.

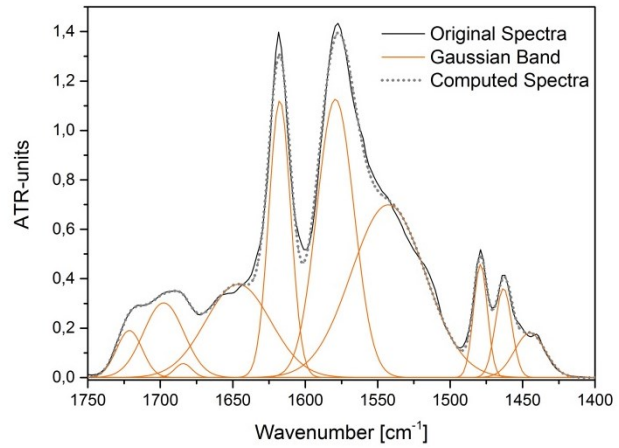

Figure 7: *Deconvolved spectrum in the carbonyl region (1750 cm^{-1} to 1400 cm^{-1}). The original spectrum was recorded after 45 min of reaction time.*

The deconvolution was conducted for all of the 19 experiments at a reaction time of 45 min. The obtained areas of the fitted Gaussian curves for the H-bonded urethane at ~1700 cm-1 as a reference for the formation of polyurethane and the urea carbonyl band at ~1620 cm-1 as a reference for the blowing reaction were selected as response signals for the design of experiments. The values of the proposed models calculated by the ANOVA are listed in Table 3. For the polyurethane response a quadratic model and for the polyurea response a reduced model showed the best result.

The polyurethane as well as the polyurea give a significant model (p-value < 0.05). The Lack of fit is not significant (p-value > 0.05).

Table 3: ANOVA values of the selected models created by the design of experiment.

Response	Polyurethane	Polyurea
Model (p-value)	0.0001	0.0001
Lack of Fit (p-value)	0.0926	0.0625
C.V. (%)	19.61	22.33
R-squared	0.9654	0.8227
Adj. R-squared	0.9307	0.7721
Pred. R-squared	0.7312	0.6440

The coefficient of variance of the models (C.V.) is as large as 22.33 % which should be considered problematic. Moreover, the R-squared, adjusted R-squared as well as the predicted R-squared do not promise a conclusive model. In order to describe qualitatively trends between the ink formulations, we concluded that the data is still satisfactory. Figure 8 shows the hypersurfaces for the obtained models. High concentrations of the iron(III)-chloride catalysts yield in high integrals of the corresponding carbonyl vibration at 1700 cm^{-1}. The influence of the concentration of the blowing catalyst BL-11 was neglected due to only small changes and the large error of the model.

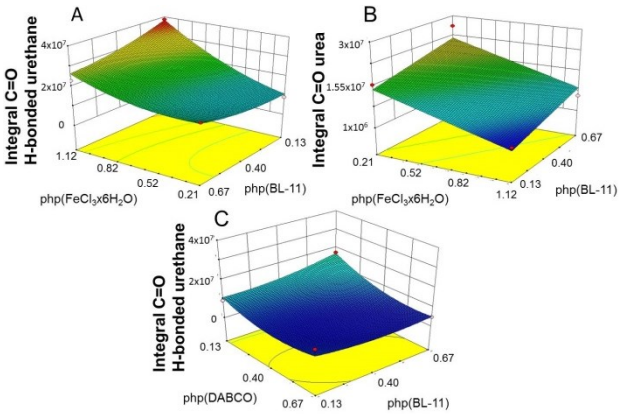

Figure 8: *Hypersurfaces of the obtained models using the DoE. The ordinate displays the obtained integral from the curve fit plotted against the catalytic concentration (parts per hundred polyol) on the abscissa. (A) Polyurethane model at low concentration of DABCO. (B) Polyurea model at low concentration of DABCO. (C) Polyurethane model at low concentration of iron(III)-chloride.*

In Part B of Figure 8, the influence of the catalysts regarding the integral of the polyurea vibration at 1620 cm-1 is displayed. For low contents of the iron catalyst an increase of the blowing catalyst BL-11 leads to high values of the urea integral. At high contents of iron, the differences between low and high contents of BL-11 are small. An increase in iron catalyst lowers the yield for the urea vibration. Part C gives suggestions about the influence of the DABCO catalyst. At low iron contents there is no significant influence evident that higher concentrations of the DABCO catalyst lead to an increase in

urethane formation. More distinct statements were not concluded due to the high error of the models.

Manual drop deposition

As a first step towards the printing of the reactive components, a deposition of the two fluids was manually conducted using 10 µl pipets. In order to obtain a stable and non-wetting droplet, the surface of the glass substrates was silanized. The hydrophobic character of the surface was verified by a static contact angle of 107.6°±1.3° with deionized water.

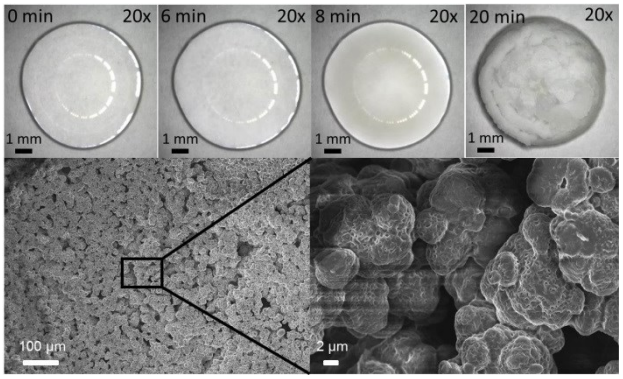

Figure 9: *(Top) microscopic pictures showing the progression of reaction of two layered droplets. (Bottom) Two SEM pictures taken from the top view of the fully reacted mixture. A cauliflower-like porous structure is visible.*

The upper part of Figure 9 shows the reaction progress with time. Compared to the preparation in bulk, the reaction seems slower as no mechanical mixing is present. After six minutes the two droplets slowly start forming turbid sections. This proceeds until the two droplets are completely turbid after eight minutes proving that mixing and the reaction of the two components takes place to some extent. After 20 minutes gas formation is visible and a cauliflower-like structure forms. The SEM images below show small pores that seem to be consisting of spheres rather than a typical pore that is well-known from water-blown foams.

Conclusion

Reactive inkjet printing of polyurethanes is a promising technology in the fields of additive manufacturing. We investigated the extension towards an even more challenging reaction forming polyurethane foams. It was found that low viscous ink formulations could be obtained using poly(ethylene glycol) 200 and glycerol ethoxylate. Combining the statistical evaluation of an experimental design with IR spectroscopy trends in influences of the catalytic mixtures were described. We conclude that a mixture of iron(III)-chloride and bis(2-dimethylaminoethyl) ether will give one a good opportunity to control the inks gelling and blowing reaction. However, 1,4-diazabicyclo[2.2.2]octane was found to influence both reactions which may be of hindrance during ink development. This knowledge can be used to specifically select ink formulations for further investigations regarding the printing and reaction behavior in a reactive inkjet printing procedure. As a first step towards the inkjet printing of reactive polyurethane foams, the manual deposition of microliter sized droplets show promising results that a layer-by-layer procedure is applicable. Yet, we conclude that there were major differences between the bulk preparation of polyurethane foams and the attempt to transfer that into an inkjet printing procedure.

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Author Biographies

Fabian Schuster *received his bachelor degree in chemical engineering for colors and coatings from the University of Applied Sciences in Esslingen, Germany, where he focused on UV-coating materials for flexible substrates. He continued his studies as a master student in the field of applied material and surface sciences at the University of Applied Sciences Esslingen & Aalen, Germany. During his Master's he focused on the pigmentation of polyurethanes for inkjet applications. Since 2015 he is working as a PhD-student at the Institute of Interfacial Process Engineering and Plasma Technology in Stuttgart, Germany in the field of inkjet applications.*

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Prof. Dr. Thomas Hirth *studied chemistry at University Karlsruhe, Germany where he received his PhD at the Institute of Physical Chemistry and Electrochemistry. From 1992 to 2007, Thomas Hirth worked at Fraunhofer Institute for Chemical Technology ICT in Pfinztal, holding different positions from postdoctoral research fellow to product manager for environmental engineering. In December 2007, he moved to Stuttgart to become the Director of Fraunhofer Institute for Interfacial Engineering and Biotechnology. Since 2008, he in addition has been professor at the University of Stuttgart and head of the Institute of Interfacial Process Engineering and Plasma Technology. In 2016 Prof. Hirth was elected as the Vice President for Innovation and International Affairs at Karlsruhe Institute of Technology KIT.*

Dr. Achim Weber*, studied chemistry at the University of Stuttgart. In 2000 he joined the Fraunhofer Institute of Interfacial Engineering & Biotechnology (IGB) as a scientist and project manager, and the Institute for Interfacial Engineering (IGVT) at the University of Stuttgart. In 2006 he became a Group Manager and since 2011 he is Deputy Head of the Department of Interfacial Engineering and Material Science at the Fraunhofer IGB. His main interest is the forming and understanding of smart, nanoscopic materials and its surfaces for applications in Pharmacy, Medicine, Environment, Material and Biotechnology.*

Dr. Monika Bach *received her Dipl. Chem. Degree from Technical University Munich, Germany, in 1988 and her PhD degree from University of Stuttgart, Germany, in 1993. After a parental leave, she joined a scientific cooperation project between Robert Bosch GmbH and University Stuttgart as Post-doctoral fellow in 1998. In 1999 she got a reentry scholarship of the University Stuttgart. In 2001 she joined the Institute for Inorganic chemistry as a project leader; University Stuttgart, Germany. In 2010 she became group leader of the Chemical-Physical Interfaces group at the Institute for Interfacial Engineering and Plasma Technology IGVP; University Stuttgart, Germany. Her major research subjects are biomaterial, biomimetic functional layers, core-shell nanoparticles, composite materials, nano- and micro-structured biofunctional surfaces.*