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The bakery of high-end sorption carbons: Sugar-urea doughs as processable precursors for functional carbons

Regina Rothe, Markus Antonietti, Nina Fechler*

Max Planck Institute of Colloids and Interfaces, Department of Colloid Chemistry, Research Campus Golm, 14424 Potsdam , Germany; *e-mail: nina.fechler@mpikg.mpg.de

Abstract

Making use of phenomena usually found in food industry, a scalable, safe and cheap synthesis method for processable and functional porous carbons is presented. By using simple sugars in combination with urea, low melting liquids can be formed which are also stable at room temperature without re-crystallization. Due to the low vapor pressure, these melts can be easily stored and handled for further use as carbon precursors. The resulting carbon materials possess high nitrogen content and are obtained in high yield. The liquid precursor state also allows for the addition of further substances, and highly porous carbon monoliths can be made by the introduction of a salt/fiber mixture. Here, cellulose is found to serve as efficient additive which results in processable viscoelastic doughs without altering the final carbon properties. This is of special importance with regard to industrial processes as, compared to standard carbonization, it becomes possible to handle intermediates in a "green body" fashion. Eventually, this allows for decoupling materials supply and processing, comparable to pre-baked bread rolls.

These carbon cookies are stable against oxidation and are shown to be highly suitable as sorption material which is demonstrated by dye-removal from aqueous solution.

Introduction

The amorphous state of sugars is omnipresent within the pharmaceutical and food industry as it is well processable, cheap, available and natural.[1] The amorphous state of sugar in pharmacy is deliberately produced by standard processes to effectively encapsulate, stabilize, and release active materials.[2-4] Carbohydrates are indeed good glass-formers and have comparably high glass transition temperatures, as we know e.g. from candy.[5] Often, the disaccharides sucrose and trehalose are used as they have glass transitions of 319 K or 368 K, while glucose as a typical monosaccharide reveals a somewhat lower value of T_g = 295 K.[6]

Less scientific, candy floss is presumably one of the earliest encounters of young scientists with this glassy sugar state, also illustrating how to use the plasticity and processability of glasses creating value and new effects. Sugar glass, also called breakaway glass, is used to simulate glass in movies, photographs and plays, with the mechanical weakness making it an excellent choice for stunts. The making of Candy floss from sugar can be compared to glass manufacturing. Here, upon the dissolution of sugar in water and heat treatment to at least the "hard crack" stage (approx. at 150 °C), a sugar glass is formed with only minor amounts of water. Practically, the addition of starch or corn syrup prevents sugar recrystallization. It is noted that all the ingredients for these technologies are cheap, scalable, and sustainable, also with respect to the processes and machines to handle the glasses. Eventually, the well-developed engineering of food allows for prices well below ordinary lab ware.

The transformation of sugars into carbons by extended baking is also a common, however, often not intended experience. In Germany, activated carbon was even called "Zuckerkohle" / "sugar charcoal", as the first sorption carbons for medical purposes were made from sugar. This material is comparably clean, poor in foreign elements, cheap, and results in high carbonization yields.[7] We believe from a synthesis view, a significant processing advantage in carbonization can be realized by applying glassy or liquid sugar precursor mixtures. As they possess little or no volatility, they can be regarded to be similar to ionic liquids or deep eutectic mixtures.[8, 9, 10] Because of the fluidity, shaping or coating can be applied. The same is true for structure-directing agents which can be introduced based on polarity and miscibility with the liquid precursor. The use of liquid precursors furthermore allows for convenient addition of co-monomers and dopants, such as nitrogen and other heteroatoms.[11] In a previous paper, we described the synthesis of heteroatom-doped nanostructured carbons based on supramolecularly pre-organized, deep eutectic precursor mixtures composed of phenols/ketones and urea.[12] Here, the processability and homogeneity of liquids could be combined with the well-defined structure of pre-aligned liquid crystals. This complemented other works using DES for carbon synthesis which, however, previously resulted in isotropic precursor mixtures and carbons of comparably low nitrogen content.[13]

In the present contribution, we want to combine the glassy carbohydrate approach with the eutectic-urea-doping. This then forms a solvent-poor carbon precursor in liquid state suitable for further processing. As it will be shown, urea is able to slightly lower the glass transition of the sugars, which is already quite low, but greatly improves processability and stability of the glassy state without re-crystallization as such. When heated, also new condensation chemistry is pre-coded. Here, the sugar /urea reaction is well known in food industry as non-enzymatic browning and is for instance applied for the browning of pretzels (also known from roasting of coffee, cacao, cereals, but then at higher

temperatures). This sugar/urea liquid is then combined with $ZnCl_2 * 1 H_2O$ as a porogen and cellulose as a processing agent, leading to a simple, convenient, and very efficient "baking-approach" towards heteroatom-doped microporous carbon monoliths. While the saccharides serve as a carbon precursor, urea does not only act as a source of doping atoms, but also cross-links the carbohydrates before carbonization to a green body which allows carbonization without further melting, i.e. preservation of shape.

Results and discussion

In the first step, the carbohydrate, here Fructose, Glucose, Xylose, or Galactose, was mixed with urea. This powder mixture was then gently heated under continuous stirring to elevated temperatures (90-135 °C) to form a clear melt. Even when this liquid was cooled down again to room temperature, it remained a clear, viscous liquid (Figure 1). This observation hints to the successful suppression of sugar or urea recrystallization, i.e. the formation of a eutectic melt. These honey-like liquids can then be pre-fabricated and stored on the kilogram scale for further processing.

Figure 1: a) Liquid monosaccharide-urea melt from Glucose.

In order to support the suppression of recrystallization, differential scanning calorimetry (DSC) data were collected in dependence on the composition of the glass mixture (Table 1). DSC further reveals the absence of crystallization peaks as well as the location of the glass transition. Taking for instance glucose, the addition of urea lowers the glass transition by about 25 K, i.e. the processing window is expanded, and the viscosity at room temperature is remarkably lowered mainly reflecting the distance to glass transition.

Table 1 Composition, melting points and yields of different sugar-urea mixtures and carbon composition after 800 °C.

Sugar : Urea	Molar ratio	Melting point	N [wt%]	C [wt%]	H [wt%]	Yield [wt%]	Carbon Yield [wt%]
Fructose	1 : 1	87.8	12,95	75.13	1.65	28.76	61.73
Fructose	1 : 1.5	85.15	14.63	74.75	1.84	23.30	52.25
Fructose	1 : 4	98.3	15,91	71.65	1.72	21.33	53.49
Glucose	1 : 1.5	74.3	13.37	76.13	1.50	23.62	53.94
Glucose	1 : 3	104.3	14.49	75.67	1.69	19.15	48.30
Xylose	1 : 1.5	101.7	14.70	75,14	1.71	22.44	50.58
Galactose	1 : 1	113.3	12.62	76.98	1.69	28.21	62.04
Galactose	1 : 1.5	111.3	14.47	75.35	1.74	26.33	59.52
Galactose	1 : 4	111.7	14.83	72.32	1.56	20.83	52.72

From the chosen systems, galactose and fructose show the highest glass transition, while xylose shows the lowest. As typical for the glass transition as such, mixtures of two monosaccharides do not result in a further lowering of T_g, but only further inhibits crystallization. Reaching a molar sugar : urea ratio of 1 : 2, slow crystallization of urea from the glassy melt is observed, which means that under these conditions processing of the sample has to be faster or elevated temperatures need to be applied.

These melts were then used for carbon synthesis and therefore heated in crucibles to the desired temperature. Here, exemplarily a carbonization temperature of 800 °C is chosen. All samples derived from the viscous melts turn into silver-shiny, fluffy solids, the metallic appearance already hinting to the special characteristics of carbons with structural nitrogen heteroatoms such as increased electron densities and electrical conductivity. Elemental analysis indeed shows that from all sugar-urea mixtures, carbons with high nitrogen content can be obtained in good yield (Table 1). As all carbon compositions are similar, the synthesis can be considered to be applicable to a rather broad range of natural educts. It is noted, that the final nitrogen content in the carbon depends on the accessibility of functional groups inside the precursor mixture, i.e. once an optimal ratio is reached, the nitrogen content remains comparable. Besides nitrogen, the samples also contain oxygen amounts due to defect and termination sites. As combustion even in oxygen atmosphere only results in incomplete combustion which is typical for this type of carbon material, the residual oxygen is estimated to be around 10 wt%.

The liquid state of the precursors allows for convenient mixing with further ingredients. As the bulk carbons were found to be non-porous by nitrogen sorption, salt was added into the process in order to introduce pores.[14] Here, to the sugar-urea liquid, a mixture of $ZnCl_2 * 1 H_2O$ is added as a porogen for pore formation within the approach of salt templating (**Figure SI-1**). It is important to mix both liquids – glucose/urea and $ZnCl_2*H_2O$/urea – both heated to 50-60°C as this allows for a homogeneous mixture. At high urea concentrations, homogeneous mixtures can be ensured by mixing additional urea also to the $ZnCl_2*H_2O$ solution at 90 °C. In the following, the porous samples were prepared with a glucose-urea mixture. Firstly, a mixture of glucose-urea with a molar ratio of 1 : 1.5 was prepared to give a liquid phase. Secondly, a separate solution of $ZnCl_2*H_2O$ was prepared from $ZnCl_2$ salt and H_2O (molar ratio 1:1). To the latter, the corresponding amount of urea was added in order to reach concentrations of urea above the eutectic point. Subsequently both liquids were mixed at 60 °C.

In order to obtain the carbon, this dough is pre-baked at 100 °C, which can indeed be done in an ordinary oven. This results in the effective cross-linking of the sugars with urea under partial release of CO_2 and H_2O which transfers the viscoelastic dough into a cross-linked polymeric material. This is followed by the comparison of FT-IR measurements of the pure ingredients and the resulting baked dough (Figure 2).
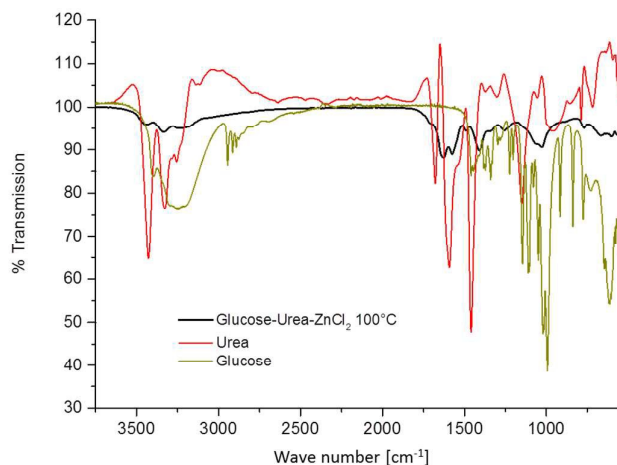

Figure 2: IR spectra of glucose (green), urea (red) and the eutectic mixture of glucose-urea-zinc chloride baked at 100°C for 1 hour (black).

Compared to the educts urea and glucose, the melt heated to 100 °C in the presence of salt already reveals a very different FT-IR pattern. The characteristic OH-vibrations of glucose vanish while the amine $\rho(NH_2)$- and $\delta(NH_2)$-vibrations of urea at 1150 cm^{-1} and 1674 cm^{-1} as well as the $v(NH_2)$-vibrations of urea at 3257 cm^{-1} and 3427 cm^{-1}, are shifted during the condensation with

glucose. This indicates the chemical reaction of urea and sugar already at comparatively low temperatures to form a cross-linked intermediate polymer which is essential for the efficient formation of carbon materials in the next steps.

Afterwards, this baked precursor dough was carbonized in nitrogen atmosphere at 500 °C, 700 °C, and 800 °C respectively. Finally, the porogen salt was removed by washing with water and diluted acid. After simple vacuum-drying, the carbon appears as a black monolith in high yield up to over 60% carbon yield (Table 2).

Table 2 Composition, yield and surface area of carbons obtained from baked cookies after salt removal.

Sample	Mass ratio	Carb.-temp.	N [wt%]	C [wt%]	H [wt%]	Yield [wt%]	Carbon yield [wt%]	Surface area [m²/g]
glucose-urea	1 : 1	500	17.9	68.1	3.1	23.4	53.1	-
glucose-urea-salt	1 : 1 : 2	500	13.4	61.7	3.6	16.1	33.1	941
glucose-urea-salt-cellulose_16	1 : 1 : 2	500	13.8	67.5	3.9	20.7	41.6	1090
glucose-urea-salt-cellulose_39	1 : 1 :2	500	12.9	70.0	4.1	25.4	48.6	979
glucose-urea	1 : 1	700	15.4	73.2	1.9	21.2	51.7	-
glucose-urea-salt	1 : 1 :2	700	12.3	57.9	2.2	15.2	29.3	600
glucose-urea-salt-cellulose_16	1 : 1 :2	700	13.0	67.5	3.0	20.3	40.8	641
glucose-urea-salt-cellulose_39	1 : 1 :2	700	12.4	70.1	3.1	24.7	47.4	802
glucose-urea	1 : 1	800	14.5	75.7	1.7	19.1	48.3	-

Nitrogen sorption of the final washed carbons indeed reveals high porosity already at relatively low carbonization temperatures representatively shown for carbons made at 500 °C (941 m^2g^{-1}) and

700 °C (600 m^{2}g^{-1}) and supporting the idea of a well-developed, stiff aromatic framework of N-containing heterocycles. (Figure 3 a). It is noted that the surface area of the carbon made at lower temperatures is higher. This is due to a higher amount of micropores in the carbon made at 500 °C as revealed by the analysis of the pore size distribution (Figure 3 b). Generally, the high nitrogen uptake of this carbon is a remarkable result as the introduction of pores in such resins is usually challenging due to a comparably soft polymer structure. The relatively high nitrogen content is an essential functional property (Table 2), too. Usually, there is a trade-off between a high surface area commonly derived at higher carbonization temperatures and a high nitrogen content which is normally obtained at lower carbonization temperatures. It is worth to comment that indeed weight-wise the reaction is practically completed at 500 °C, due to the described elimination schemes, while further heating certainly improves aromatization and rearrangement of the condensed aromatics towards extended 2d-sheets, however without significant change in mass and nitrogen at higher carbonization temperatures: This hints to the stable incorporation of nitrogen sites into the aromatic resin already at 500 °C. It is noted, that the synthesis in the presence of the zinc salt results in an increase of the oxygen content of about 5wt% due to carbothermal reduction. Eventually, the synthesis presented herein also allows for the preparation of resin-like materials with both, high porosity and high functionality which are important with regard to applications.
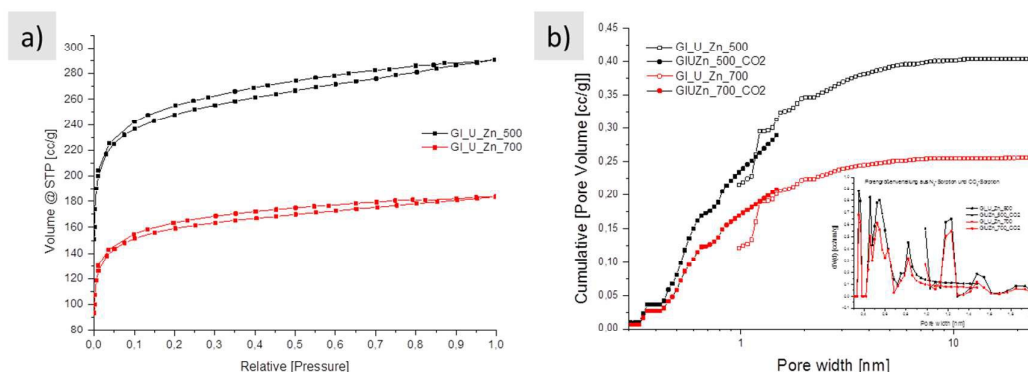

Figure 3: **a)** Nitrogen sorption isotherms and **b)** pores size distribution from nitrogen (open circles) and CO$_2$ (filled circles) sorption of carbons made from glucose, urea and ZnCl$_2$ carbonizetd at 500 °C (black) and 700 °C (red).

In order to give the precursor mixture more elastic cohesion, i.e. to allow for more shape-retaining dough and easier processing e.g. into beads or solid pellets (cookies), cellulose was added as a binder, keeping a constant ratio of the remaining ingredients (Figure 4). Here, the viscosity can be easily tuned by the amount of added cellulose fibers, again a fact well known from food processing.

Figure 4 Doughs from glucose–urea–$ZnCl_2$/H_2O + 39% Cellulose before and after baking **a)** viscoelastic pellets of the dough, **b)** pellets pre-baked at 100°C for 3 hours, the green body, and **c)** carbonaceous pellets made at 500°C in nitrogen atmosphere.

As the resulting objects are indistinguishable from food with regard to appearance and haptics, we call them "cookies" and after final carbonization "carbon cookies", respectively (Figure 4 b, c). The intermediate can be stored and afterwards further carbonized at high temperatures in different surroundings. This is of special interest as conventional impregnation methods require immediate further processing which is difficult with regard to scaled processes. This stability for transport and storage is attributed to the ability of cellulose to gel the system by secondary interactions.

The baked pellets were then carbonized in a nitrogen oven which resulted in black monoliths (Figure 4 c). The macroscopic size of the final carbon slightly shrinks compared to the initial raw cookie, however, the monolithic form is retained throughout the process. After washing with simple diluted hydrochloric acid and water, also here nitrogen doped carbons with high porosity are obtained (Figure 5).
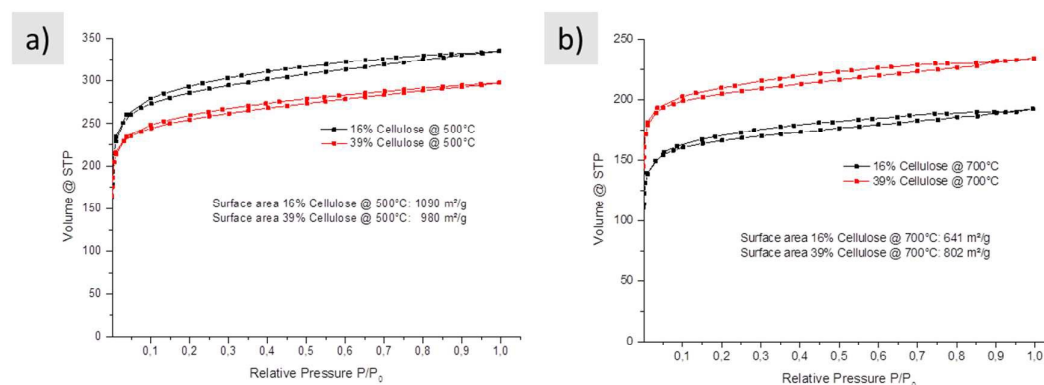

Figure 5: Nitrogen sorption of carbons made from glucose-urea-salt with different amounts of cellulose (black 16% and reed 39 %) carbonized at **a)** 500°C and **b)** 700 °C in nitrogen after washing.

These results reveal high surface areas and microporous materials also for the carbons which were prepared with the additional cellulose filler. This is in agreement with the texture observed in Scanning Electron Microscopy (SEM), which reveal smooths and homogeneous carbon structures (Figure SI-3). The comparison to the carbons prepared without additional cellulose shows that the porosity and composition of the final carbon prepared with the filler did not change significantly, which could be somewhat expected. The cellulose indeed mainly serves as an additive to address current processing issues rather than being a main part of the carbon material.

It is a special feature of nitrogen-containing carbons that the heteroatom incorporation can improve the oxidation stability of carbon materials.[15, 16] Here, our carbon cookie made at a low carbonization temperature of 500 °C and with 39 wt% of cellulose was then used to test the thermal stability of the final material under harsh conditions. For this, the cookie was repeatedly placed into the flame of a butane/propane (70 : 30) gas burner (Figure 6a, video SI). Even after several cycles reaching also a red blaze in the central flame (~800 °C), the carbon remained its macroscopic shape (24% weight loss after 6 heating/cooling cycles à 3 s). Considering the very low carbonization temperature and high porosity, this is a remarkable result keeping in mind the usually fast ignition of untreated cotton or paper (Cellulose). This hints to the efficient and homogeneous mixing of the liquid precursors leading to water elimination and N-heterocycle formation already at comparably low temperatures. This essentially is a reflection of the low mass loss revealed by TGA measurement which is mainly due to the gases released from cellulose and/or carbon which otherwise promote the flaming, while the release of water under formation of an oxidation stable N-doped carbon causes the observed fire inertness (Figure SI-4). Of course this makes the material suitable as a catalyst support or for high temperature thermal insulation based on the micropore content.
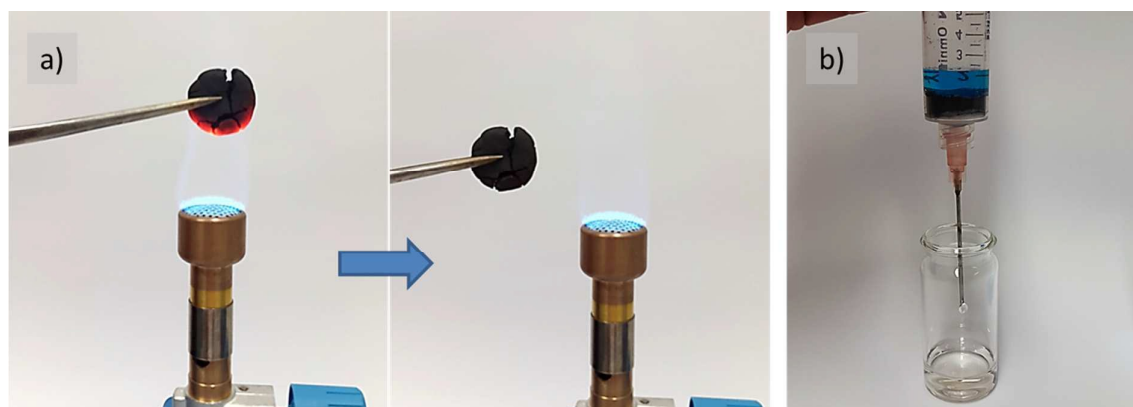

Figure 6: a) Images of the nitrogen-doped carbon cookie made from glucose-urea-salt with 39 wt% of cellulose carbonized at 500°C **a)** monolith during repeated firing cycles using a butane/propane gas burner and **b)** powder during sorption of methylene blue dye from aqueous solution.

In a further test, the accessibility and functional character of the pore system of the carbons was exemplified by dye sorption using methylene blue as a comparably large model sorbent for visualization (Figure 6b, video SI). For this, the carbon was placed in a syringe followed by addition of the dye solution. As can be observed from the colorless liquid at the syringe tip after squeezing through the carbon sorbent, even the comparably short contact time during flow allows for an efficient dye removal. This is attributed to the structural nitrogen content which increases the sorption strength of the carbon surface and the good accessibility of the pore system created by the aqueous salt porogen removal during synthesis which allows for the preservation of the initial cookie shape.

Conclusion

In this work, the synthesis of porous nitrogen-doped carbon materials using processable sugar glasses as precursor system was presented. It was shown that the addition of urea to several sugars substantially reduces the melting point of the solid mixtures and even can suppress recrystallization. All substances are cheap, safe and available in big quantities which allows for an efficient carbon synthesis. Through the addition of salt and cellulose as porogen and structural filler, respectively, it was furthermore possible to prepare storable green bodies which are an important aspect with regard to industrial processes. Carbonization of these intermediates resulted then in functional porous carbons in form of monoliths, i.e. "carbon cookies". These materials were then demonstrated to be stable against oxidation and can serve as adsorbent in aqueous media. As the synthesis approach is modularly adjustable, scalable, resource efficient and compatible with standard industry equipment, we believe that it allows for further steps in fields where high amounts of porous and functional carbons such as energy storage and catalysis are needed.

Acknowledgement

Materials and Methods

The water-free sugars glucose, fructose, galactose, xylose were obtained from ROTH. Urea, zinc chloride and Cellulose were purchased from Sigma-Aldrich. All materials were used without further purification.

Synthesis

Non-Porous carbon

From a powder mixture of sugar (glucose, fructose, xylose or galactose) and urea (molar ratio 1:1.5) a clear and slightly yellow viscous liquid is formed by heating it in an open glass vial using an oil bath at 90-130 °C. This liquid can be cooled down and later heated again. For the synthesis of the final carbon, this liquid is then directly heated in nitrogen atmosphere to result in nitrogen-doped carbon Heating program: 30 min purging at room temperature, heating rate 2.5 K min^{-1}, final temperature 500 °C and 700 °C, respectively, hold for 1 h.

Porous carbon

In order to obtain porous nitrogen-doped carbon, the sugar urea liquid can be mixed with zinc chloride as porogen. Here, zinc chloride is separately mixed with an equimolar amount of water at 100°C until a homogeneous mixture is obtained. As the amount of urea in the initial sugar-urea mixture is limited, additional urea for more nitrogen in the final carbon can be added to the zinc solution. For this, the zinc chloride water mixture is cooled down to 90 °C. Afterwards, urea is added under continuous stirring. The addition leads to a clear colorless solution. Afterwards, both liquids, the sugar-urea and zinc chloride-water(-urea) mixtures, are mixed together at 60-80°C. The combined mixture is then first baked at 100°C for 1 hour. This green body can then be heated at elevated temperatures in a nitrogen oven following the heating program from above. Finally, the zinc salt is removed by simple washing with water and 1 M hydrochloric acid (500 mL) overnight and the porous carbon is recovered by vacuum filtration.

Carbon-monolith

For the preparation of storable pre-pellets and final monolithic carbons, cellulose is added to the mixture of glucose-urea-zinc chloride (relative amounts are kept constant). After mixing, this bulk is poured into a petri dish or formed to pellets which are then dried at 100°C for 3 hours. Afterwards, these pellets can be stored or carbonized in nitrogen atmosphere at the desired temperature (500°C – 800°C). For the washing process the carbonized samples are grinded in a mortar and wash with 1 M hydrochloric acid for 24 hours. After filtration, a second wash is made with water followed by the

final drying step in vacuum at 60°C. Alternatively for the monolith, the pellets were soaked in hydrochloric acid and water respectively, followed by drying in vacuum.

Characterization

Elemental analysis was performed as combustion analysis using a Vario Micro device. Scanning electron microscopy (SEM) was performed using a Gemini Leo-1550 instrument after sputtering with platinum. Fourier transformed infrared spectra (FT-IR) were obtained with a Varian 1000 FT-IR spectrometer with an attenuated total reflection setup. Nitrogen sorption measurements were accomplished with N_2 at 77 K and CO_2 at 273 K, 284 K and 302 K (after degassing the samples at 150°C under vacuum for 20 h) using a Quantachrome Quadrasorb SI porosimeter. The apparent surface area was calculated from the nitrogen sorption measurements by applying the Brunauer–Emmett–Teller model to the isotherm data points of the adsorption branch for p/p0 < 0.3 (BET area). The pore volume was calculated from the obtained physisorption data with the program QuadraWin (Version 5.11), where the mesopore volume was calculated by subtracting the micropore volume from the total pore volume. The pore size distribution, the cumulative pore volume and the average pore size were also determined with the program QuadraWin (version 5.11) by using the quenched solid functional theory (QSDFT) for slit/ cylindrical pores applied on the nitrogen adsorption isotherm.

For the fire-retardant experiment, the carbon monolith prepared from glucose-urea-zinc and 39wt% after a carbonization temperature of 500 °C was placed repeatedly into the flame of a butane/propane gas burner and weighted before and after. For the dye sorption test, the same carbon was milled and used in combination with an aqueous methylene blue solution (10 mg L^{-1}). This carbon was then placed into a syringe equipped with a piece of cellulose filter paper followed by addition of the dye solution and dropwise squeezed through the carbon.

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Journal of Materials Chemistry A Accepted Manuscript

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Table of contents entry

The bakery of high-end sorption carbons: Sugar-urea doughs as processable precursors for functional carbons

Regina Rothe, Markus Antonietti, Nina Fechler*

Max Planck Institute of Colloids and Interfaces, Department of Colloid Chemistry, Research Campus Golm, 14424 Potsdam , Germany; e-mail: nina.fechler@mpikg.mpg.de

Porous nitrogen-doped carbon monoliths from processable doughs of liquid sugar-urea mixtures with salt and cellulose as porogen and filler, respectively.