Final report

1.1 Project details

Project title	CleanTechBlock 2
Project identification (pro- gram abbrev. and file)	64015-0018
Name of the programme which has funded the project	EUDP
Project managing com- pany/institution (name and address)	Gråsten Teglværk A/S
Project partners	Aalborg Universitet for Chemistry and Bio (AAU-SC), Josef Stefan Institute, Slovenia (JSI), Aalborg Universitet Dpt. For Civil Engineering (AAU-CE)
CVR (central business register)	40294619
Date for submission	03.03.2019

1.2 Short description of project objective and results

På Dansk: Formålet med dette projekt var at udvikle skumlgas med en termisk ledningsevne på 37 mW/(mK) and at demonstrere at skumglasset kan opskaleres til 1:1 størrelse. Disse mål blev realiseret ved at vi opnåede bedre forståelse for mekanismerne bag opskumningen og for de bagvedliggende faktorer, som styrer varmetransporten i skumglasset. Store blokke af skumglas blev produceret med pilotanlæg og brugt til at opstille en CleanTechBlock (CTB) testmur for at kunne dokumentere installationsprocedure af den samlede CTB vægløsning. Resultaterne blev brugt til at designe et demonstrationshus i rigtig størrelse. Konstruktions-processen blev dokumenteret og analyseret. Huset vil blive brugt til at fremvise CTB vægløsningen overfor arkitekter og kunder. Resultaterne fra dette projekt blev desuden sammen med en markedsanalyse brugt til at justere forretningsplanen for CTB løsningen.

In English: The project objectives were to develop a foam glass with a thermal conductivity of 37 mW/(m·K) and demonstrate its up-scaling feasibility by preparation of 1:1-sized foam glass blocks. These objectives were achieved by gaining new knowledge on the foaming mechanism and on the different contributions to the effective thermal conductivity. The demo foam glass blocks were used to prepare a CleanTechBlock (CTB) test wall in order to document the insulation performance of the CTB wall solution. The results were used in the design of a demonstration full-sized house. The construction process was documented and analysed. The house will be used to showcase the CTB solution to architects and customers. Furthermore, a market analysis was performed and business plan was updated with the input of the project results.

1.3 Executive summary

The CleanTechBlock 2 project demonstrates the upscaling, production feasibility, market potential and construction process for a new sustainable building block. The CleanTechBlock (CTB) is a multifunctional composite of a foam glass core as insulation material and clay brick

shells as structural and aesthetic component. In the project a pressing technology was developed that allows production of clay bricks in a new large size without flaws. For the efficient insulation performance of CTB we developed a new process for preparation of foam glass with improved insulation properties by a less energy-intensive process. The new process was developed based on the understanding of the foaming mechanism. This knowledge enabled us to tune the foaming process and achieve a low density (90–110 kg/m³) and high content of closed porosity (82–99 %) filled with CO₂ (65–95 %) exhibiting thermal conductivity as low as 37 mW/(m·K) measured by a Heat Flow Meter at 10 °C.

We demonstrated the upscaling potential of foam glass by preparing foam glass blocks in 1:1 size ($50 \times 10 \times 45$ cm). The foaming was performed in a large chamber furnace with sufficient atmosphere and temperature control. The filling of the steel mould had to be adjusted to allow a preparation of foam glass without larger holes. The optimized large blocks exhibited a density of 115 kg/m³. 20 blocks were produced for preparation of a test CTB wall, which was tested for insulation performance. The average density of the blocks in the wall was 135 kg/m³ and thermal conductivity 48 mW/(m·K) measured with a Guarded Hot Plate. The increase of the thermal conductivity in comparison the small samples was related to the elongation of the cells in the large plane of the samples. The elongation formed during the foaming process due to specific conditions for the preparation of the large foam glass blocks. The effect of the cell elongation on the thermal conductivity is expected, but the size of this effect is much larger than predicted.

The test CTB wall was assembled from foam glass blocks and clay brick shells prepared by project partners. The testing in a Guarded Hot Box revealed a thermal conductivity of 53 $mW/(m\cdot K)$. The increase is related to the imperfections in the wall assembly as well as some larger cells present in the foam glass blocks. The construction process of CTB was assessed by constructing a full-size house using clay shells prepared in a pilot production process and commercially available foam glass blocks. The building is used to gather responses from architects, engineers and customers. The market potential of the CTB solution was analysed and the business plan was updated with the input from the construction process, market analysis, and stakeholder response.

The research organizations will exploit the results of this project for teaching and preparation of follow-up projects. One follow-up project was granted and its results are upgrading the results of this project financed by EUDP as well as increasing the potential of industrial exploitation of the CTB. Besides the scientifically relevant projects, researchers and Gråsten Teglværk discuss possibilities for collaboration with companies designing foam glass production lines as well as companies producing foam glass in order to speed-up the utilization of the results for commercial benefits.

1.4 Project objectives

The overall goal of the CleanTechBlock 2 (CTB) project is to demonstrate the upscaling, production feasibility, market potential and construction process for a new sustainable building block. The CleanTechBlock (CTB) is a multifunctional composite of a foam glass core as insulation material and clay brick shells as structural and aesthetic component. The first objective of this project was to improve the insulation performance of foam glass and to develop pressing technology that allows production of large size bricks without flaws. The foamed glass block and the large sized brick shells are joined together with a mineral glue to form a CTB building block. The development of foam glass was focused on the composition of the gas entrapped in the closed pores and the ability to obtain small pores with high level of closed porosity. The goal for the thermal conductivity of foam glass was set at 37 mW/(m·K), which is similar to the value for standard mineral wool used for insulation in buildings, i.e. 35 mW/(m·K).

The second objective was to demonstrate that the CTB has business potential. New foam glass is at this point produced at a laboratory scale. Experience show that there is large gap between laboratory and industrial production. To demonstrate up-scaling potential and industrial production feasibility, the foam glass needs to be prepared on a 1:1 scale in a pilot-scale furnace (the furnace was purchased with funding from Obelske Fund). Then a test wall with a surface of 1 m^2 will be prepared and tested for the insulation performance. CTB prototypes from pilot brick shell production and commercial foamed glass will be used to build a demonstration house. The house will be used to showcase the possibilities of the CTB product, measure the short construction time requirement, and to gain feedback from partners (the construction of the house is financed from Horizon 2020 SME Instrument scheme). Physical and thermal tests will be made on the walls to certify insulation ability and humidity permeability. Evaluations of the wall and house will be included in the final business case (the market potential analysis was financed by the Horizon 2020 SME Instrument scheme).

Two important risks related to the foam glass development were identified 1) ability to entrap a low-conducting CO₂ gas in the closed pores and 2) reach a low thermal conductivity ≤ 37 $mW/(m\cdot K)$. These risks were marked with a Stop/Go decision in the proposed plan. Both targets were successfully achieved thus the project continued with demonstration activities (at month 20). Initially, the development of foam glass was hindered by the small available size of the samples produced in the laboratory and related difficulties to properly measure the thermal conductivity of the samples. For these reason several instruments were tested and two new instruments were purchased. Before the sample size was increased, the measurements were performed with a transient plane source method using a HotDisk instrument. To correctly evaluate the measured values, a comparative study with commercial samples measured with a standard steady-state method using a Guarded Hot Plate (GHP) instrument and the HotDisk instrument was performed. The study showed a good performance of the HotDisk instrument but the exact values were still not precise enough. The second instrument purchased is a custom-made steady-state heat flow meter (HFM), which enables measurements according to a standardized method. The size of the laboratory samples increased to 8 cm x 8 cm x 2 cm (after final shaping) by installing a large-diameter tube furnace with atmosphere control. HFM values were in perfect agreement with the GHP measurements on large sample size. Thereafter, we were able to demonstrate the real insulation performance of the foam glass developed in the laboratory.

The demonstration of the up-scaling potential and industrial production feasibility was initiated after installing a large chamber furnace at AAU-SC. The selection of a suitable furnace was based on the size, possibility to control the atmosphere, available budget, installation space limitation, running costs and amount of required demonstration samples. The installed chamber furnace was the best option and allowed the minimum required conditions for the atmosphere control (the concentration of residual O_2 in the furnace was 0.8-1 %). Batch of 150 kg was prepared by external partner for preparation of the demonstration samples. The preparation of demo samples was highly demanding, consuming large amount of working time and resources. After long optimization process, we were able to prepare the needed amount of samples in 1:1 scale. The samples density was 130–135 kg/m³, which is more than the density of the laboratory-sized samples (100 kg/m^3) , but adequate for the demonstration. The thermal conductivity of the samples was evaluated on two scales. For the small scale characterization a piece of foam glass block was cut and measured with HFM. The measured values were 40.6-42.5 mW/(m·K) at densities of 132–140 kg/m³. These results are in full agreement with the laboratory samples, confirming the upscaling possibility. For large scale characterization a whole block was measured using GHP. These results showed a higher thermal conductivity (for about 15 %), which was in main part connected with the different orientation of the sample. Due to specific foaming conditions the cells were elongated in the in-plane direction, thus the thermal conductivity is higher in this direction. Moreover, some inhomogeneity remained in the large blocks: few larger voids, the pores

close to the surface were larger and partially open. In the case of industrial production these deficiencies are expected to be solved by the continuous automated batch preparation.

The demo 1:1 foam glass blocks were used to prepare demo 1:1 CTBs, which were used to build a test wall element. The mock-up element was tested for insulation performance at AAU-CE and the documentation on the foam glass and CTB mock-up was collected. The results show that the insulation performance decreases to $53 \text{ mW/(m \cdot K)}$, when foam glass is incorporated into the CTB wall. The increase is similar with the increase observed for a CTB test wall prepared with commercial samples (Danish National Advanced Technology Foundation – Højte-knologifonden under grant number 012-2011-3), which indicates that at least part of the increase can be related to the imperfect junctions between adjacent CTBs. Thereafter, the construction process needs to be very precise. The construction process will be developed further and assessed during construction of a demo CTB building.

The funding for the demonstration building and the market analysis was provided by the SME Instrument in the Horizon 2020 European funding scheme. The project started 1. June 2017 and will be finished in May 2019. The customer response has been positive in time of the project. We have had many inquiries from engineers, architects and other from the construction industry. Customer response will continue to be collected after the construction of the demo building. The business plan was updated with the input from the market analysis, architects and engineers' response, and the demonstration CTB house construction.

1.5 Project results and dissemination of results

According to the results described below, we conclude that the project succeeded in realizing the objectives to a great extent. The project answered most of the problems stated in the project proposal. The targeted thermal conductivity of foam glass, i.e. 37 mW/(m·K), was reached, while the best samples contain a moderate amount of CO in the closed pores (30–35 vol.%). The upscaling and the demonstration of the 1:1-sized foam glass was successful. The demonstration wall from CTB blocks produced from the demo foam glass and the demo large-sized brick shells was tested. The building of a fullsize house (using a commercial foam glass and brick shells from a pilot production), customer response and review of the business plan financed by the Horizon 2020 SME instrument will be finished in June 2019. As shown in Annex 1, the project results were disseminated through publications in scientific journals, presentations at scientific conferences, and technical reports, attaining the construction fairs, and presentations of student projects at Aalborg University. In addition, the project has led to a Ph.D. Thesis (see Annex 1).

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Task 1.6 Furnace engineering																								M6								-				1
Task 1.7 Foam glass prototype																															M3		-			1
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WP 2 Demonstration site	1																														-	-	-		-	1
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Task 2.2 Mock-up building and testing																			\top													-				1
Task 2.3 Real size house																					Г											M4				1
Task 2.4 Customer response																					1															1
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Technical milestones The technical milestones The technical milestones show at which time mechanical, thermal and chemical parameters have to be developed and optimised, before a CleanTechBlock wall solution is commercially meaningful. Commercial milestones The commercial milestones show at which time the CleanTechBlock with improved technical features has been demonstrated. The final milestone is to document that CleanTechBlock can become a business.																																				
The scientific milestones are related to publication outcome, such as Stop-Go decision #1 Decide if pilot furnace should be build. Ability to form pure CO2 i	publi n foar	icatio n gla	ons ii ss is	n inte a key	ernation / decisi	nal jo	ournal	s and	l cor	iferei	aces.																									
#2 Decide if phase 2 should start. Foam glass with low thermal cond	utivit	y (<3	7 m\	V/(m	K)) is k	ey d	ecisio	n par	ame	ter.																										

Figure 1: Gantt chart of the project implementation, objectives and milestones.

Overview of the project activities

WP1: Foam glass and brick production

<u>Task 1.1. Brick production.</u> The bricks are made in a length of 52.5 cm, a height of 10.5 cm and a thickness of 4 cm. These dimensions of the bricks present great challenges for the processing. Because when the clay dries before it is burned, the bricks becomes very unstable. We have succeeded in producing bricks with a tolerance in the straightness within 1 mm and at the edges of the brick shells are formed with a projection and a recess, conferring structural stability, insulation properties, good aesthetical appearance and making the wall construction easier and less error-prone process. The fact that the narrow glue joint is hidden implies several advantages. The glue joint is protected against water and wind, in this way increasing the time needed for the first wall maintenance from 50 years to 150 years. Moreover, the glue joint can be made of a material with any colour without being visible. Technical milestone M1 Flawless bricks was successfully achieved.

<u>Task 1.2. Closed foam structure.</u> Foaming experiments focused on improving the microstructure of the foams were conducted by modifying surface tension of glass, glass composition and addition of crystallization inhibitors. Potentially interesting compounds contain potassium, boron, calcium, zinc and phosphate. The compounds were added in relatively small amounts from 2 to 6 wt.% and showed a strong effect on the foaming process and pore microstructure. This study identified a suitable compound to tune the surface tension of the glass, thus enabling a more homogeneous pore size distribution and a higher fraction of closed pores.



Figure 2: Open-porosity content (OC) as a function of the density of the samples. The values are comparable to best commercial products for the same density and sample size.

Task 1.3. Gas composition. The method for analysing gas composition using gas chromatography was upgraded for a proper identification of the relevant gases. The results showed that for the investigated foams the major gases in the closed cells are CO_2 and CO. CO_2 has a thermal conductivity of 16 mW/(m·K) and CO 26 mW/(m·K). Thus it is crucial to adjust the foaming mechanism to secure CO₂-filled pores. Our investigation showed that the gas composition depends on the type of the carbon used. The more stable the carbon additive, the higher the content of CO₂. Moreover, we observed that for the higher cooling rates the content of CO was higher, than for the smaller cooling rates. The CO is formed at the foaming temperature where the CO is preferred gas component in the CO/CO₂ equilibrium, i.e. Boudouard equilibrium. We investigated the change of the gas composition on annealing the foam glass at around the glass transition temperature, i.e. 530°C. The CO₂ increases in general with time during low temperature heat-treatment (463–650 °C). The conversion is faster at a higher annealing temperature. Based on this investigation we considered the first Stop/Go point related to the gas composition and decided to continue with the purchase of a big furnace. The scientific milestone M6 Recipe for gas composition was achieved. However, at a later stage new samples with a very low density were prepared for which we observed some portion of CO present in the closed cells. For these samples the annealing at a moderate temperature 550 °C for 5 h failed to alter the gas composition significantly. Additional experiments revealed that this is related to the use of manganese oxide as the oxidizing agent, since pure CO_2 was detected in the samples with iron oxide as the oxidizing agent. DTA-MS studies showed that for the manganese oxide containing samples CO starts to develop at a lower temperature than in the samples with iron oxide. We identified several possibilities to tune the gas composition further, which are being investigated in a currently running project funded within European M-era.Net scheme. Despite the moderate content of CO, the last samples with the low thermal conductivity also exhibit a very low thermal conductivity as described later.



Figure 3: DTA-MS analysis of the standard composition. CO content increases with increasing temperature above 850 °C, which is related to Boudouard equilibrium.



Figure 4: Thermal conductivity of standard composition (blue curve), best commercial samples as declared (red curve) and CO_2 content in the closed pores for the standard composition (orange curve) as a function of density. Thermal conductivity was measured by a HFM at 10 °C.



Figure 5: Content of CO_2 in the closed cells as a function of annealing temperature and time for samples with 0.31 and 0.4 wt.% carbon. Typical annealing time of foam glass in industrial production is 240–480 minutes.

Task 1.4. Glass composition. The thermal conductivity of a glass is related to the composition and influences the solid conduction contribution to the effective thermal conductivity of the foam glass. In this relation we investigated the influence of various foaming additives on the thermal conductivity of solid glass. The samples were either sintered (in this case some crystalline phases were present in the glass) or melted (in this case the samples were fully amorphous). The results showed that the melted samples have a lower thermal conductivity, which was the lowest for the samples with added manganese oxide. The positive effect of manganese oxide on decreasing the thermal conductivity of solid glass was theoretically predicted. On the other hand, also the negative effect of crystallization was expected. These results clearly demonstrate the guidelines for a foam glass with improved insulation properties. Thus our research focused on preparing a crystal-free foam glass with the addition of manganese oxide as an oxidizing agent. We also investigated the influence of different cullet sources showing glass foams prepared from CRT panel glass exhibit a lower thermal conductivity than the ones prepared from flat glass, container glass, and mixtures of these three types. This difference can be attributed to the lower thermal conductivity of bulk CRT panel glass compared to the other two types. Furthermore, we performed some experiments and calculations to see how the pore structure influences the thermal conductivity.



Figure 6: The thermal conductivity ($\lambda = \lambda_{meas}$ or λ_{solid}) of the panel glass mixed with either a) MnO_2 or b) Fe_2O_3 . The thermal conductivity of the sintered samples (λ_{solid}) is corrected for the residual porosity (measured by the laser flash method at 25 °C).

<u>Task 1.5. Crushing and mixing.</u> The particle size of the waste glass and the additives have a large influence on the foaming process and homogeneity of the prepared foam glass. Our investigation was focused on identifying optimal milling and mixing conditions. The optimal particle size for waste glass was determined to be at 5–7 μ m (D50) and similar for manganese oxide, which is added just before the end of the milling process. These particle sizes are similar to the ones applied in the commercial production of foam glass with superior properties.

<u>Task 1.6. Furnace engineering</u>. In order to investigate industrial feasibility of the developed process a scale-up of the laboratory samples to the full 1:1 size was performed. In the first step we installed a tube furnace with a large tube diameter, which enables the preparation of samples with final size 8 cm x 8 cm x 2 cm. This sample size was crucial for the proper evaluation of the thermal conductivity of the samples. The furnace enables very good control over the atmosphere, temperature and heating/cooling rates. For the demonstration 1:1 sized samples we purchased a large chamber furnace that also enabled sufficient control over the atmosphere, temperature and heating/cooling rates.



Figure 7: Interior of the large furnace for production of 1:1 sized foam glasses.

Second Stop/Go decision was related to the ability to achieve a thermal conductivity value of 37 mW/(m·K). In order to confirm thermal conductivity values of the prepared foam glasses a custom made HFM for sample size 8 cm x 8 cm x 2 cm was installed. For comparison reasons large commercial samples were purchased and initially tested with a GHP apparatus. Then the samples were cut and tested also with the HFM. The results were in perfect agreement. Thus we were able to reliably test the insulation performance of our samples. The thermal conductivity of 37 mW/(m·K) was obtained for sample with a density of 95 kg/m³. These properties are very similar to the best commercial product, however, in our case they were achieved by a direct foaming of waste glass without the re-melting step to adjust the composition as used in commercial production. Hence, we reduced the energy consumption of the production process of foam glass.



Figure 8: The thermal conductivity of the standard composition as a function of the density. The targeted thermal conductivity of 37 mW/(m·K) was reached (measured at 10 °C by a HFM). The saturation of the thermal conductivity values below 110 kg/m3 is connected with the increasing CO content (Fig. 4) and open porosity (Fig. 2).

<u>Task 1.7. Foam glass prototype.</u> Foam glass prototype was prepared in the large chamber furnace from a large batch of waste glass–foaming additives mixture. Two batches were prepared, a 120-kg batch of a standard mixture and a 30-kg batch of a modified mixture. The batches arrived to Aalborg University in five barrels. The barrels were sealed to avoid take up of moisture from the surroundings. The upscaling was completed by increase the sample size step-wise. A total of forty large size glass foams were made. More than thirty trials were made to optimise preparation procedure, gas- and temperature program for low density and homogenous pore structure. The furnace is equipped with a fan in order to have a good temperature distribution inside and the heat-treatment was carried out in a low oxygen atmosphere by purging through the furnace chamber with nitrogen. An oxygen sensor was used at 25-100 °C to check the oxygen concentration during the purging program. The temperature was checked with up to four external thermocouples to assess the influence of sample insertion on the temperature distribution inside the chamber.



Figure 9: Barrels with powder mixture.

A stainless steel mould was placed on a perforated stainless steel plate. The plate was placed on the bottom shelf. The stainless steel mould is equipped with ceramic fibres on the side walls and on the stainless steel base plate. The ceramic fibre mats prevent the viscous glass foam sticking to the stainless steel. The carbon sprinkled around the batch material acts as an oxidising agent, consuming remaining gaseous oxygen at >400 °C. Placing a lid on top of the mould was fond useful during the optimisation process. The lid covered approx. 85% of the top. The lid also had ceramic fibre to avoid sticking of glass melt directly on the lid.



Figure 10: left – Mould with a newly prepared green pellet. The wood scaffold is used for compacting a rectangular pellet. Carbon powder is spread on the bottom side. Right – Lid for the mould.

The pellet was hand-pressed from the powder mixture. Several layers of powder are needed to ensure high compactness. For a 5.5 kg sample, a minimum of sixteen layers are needed. Each layer consists of maximum 500 g. When deviating from this rule, elongated holes inside the glass foam appears (in Danish: Lunker). The mould had a dimension of 53x48 cm. The pellet for the final large size foam was approximately 25x22x10 cm (LxWxH). It was found that the pellet width and length must be tuned in relation to the mould size. If the pellet dimensions are too large, the bottom side lift itself from the bottom, creating a cavity underneath the form. This effect is called "wormhole effect", since the caving can lead to holes with a penetration depth equivalent to more than 50% of the foam height. If the pellet size is too small, the foaming melt will not fill the entire mould.

The temperature program consist of three steps. The first step (I) ensures the exchange of air with N_2 . The gas transport from the pellet is slow, since it is based on diffusion. The heating rate is initially high in order to increase the diffusion rate of the gaseous species. This increases the exchange rate of air with N_2 from the interior of the pellet to the surroundings. Carbon oxidises fast above 400 °C, therefore the temperature was kept at 400 °C or below for at least 2 hours. In step (II) the glass powder sinters a closed body. From approx. 700 °C the foaming proceeds. It was found that the heating rate must be relatively low, to ensure that no large temperature gradient was formed from the sample interior to the surface. This step ends with a fast cooling to freeze-in the foam structure. In step (III) the glass foam is annealed to avoid any stress in the final glass foam. The annealing program follows an annealing program of commercial glass foams. Finally the temperature is cooled down to room temperature.

It was observed that the glass foam forms a skin on the surface. This skin could rupture during foaming and cause very large fissures in the foam. Placing a lid on top of the mould minimises this damage. The lid also helped divided the foam more evenly in the mould. Without a lid the foam would rise in the middle of the foam and not filling the corners of the mould sufficiently.



Figure 11: Glass foams just after heat-treatment. The purple crust on the surface of the samples is formed due to presence of a small amount of residual oxygen in the furnace atmosphere during heating.

The optimized glass foam using the above mentioned sample preparation procedure, mould design and heat-treatment program led to a glass foam with a density of 116 kg/m^3 with homogeneous pore structure. The expected gas composition was > 90% CO₂ and a thermal conductivity value of 38.5 W/(mK). The glass foams shipped to the AAU-CE (Department of Civil Engineering, Aalborg University) for shaping and construction of a mock-up. The foam glass blocks have on average a density of $130-135 \text{ kg/m}^3$ and thermal conductivity of 40.6-42.5 mW/(m·K) in the direction perpendicular to the large surface plane.

WP2 Demonstration site

<u>Task 2.1. Documentation of foam glass.</u> Documentation on the laboratory-sized foam glass and the demonstration 1:1 size foam glass was collected. The laboratory samples exhibit a thermal conductivity of 37–37.4 mW/(m·K) at a density of 90–105 kg/m³ and 41 mW/(m·K) at a density of 135 kg/m³ (measured using the HFM at average sample temperature of 10 °C). The demonstration samples measured with the HFM show a thermal conductivity of 40.6–42.5 mW/(m·K) at a density of 132–140 kg/m³, which is in accordance with the laboratory samples. These values were obtained for the heat flow perpendicular to the large plane. The values for the heat flow parallel to the large plane were 45.6–47 mW/(m·K) at a density of 132–140 kg/m³. Such observation is related to the elongation of the cells in the large plane due to specific conditions for the preparation of the demo samples. Since the green sample needed to be smaller than the mould, the foaming was initiated in the lateral directions, thus forming cells elongated in the in-plane direction. The effect of the cell elongation on the thermal conductivity is expected, but the size of this effect is much larger than expected. This give the opportunity to exploit the effect for decreasing the thermal conductivity of future products.

<u>Task 2.2. Mock-up building and testing</u>. The CTB test wall was prepared from the demo foam glass and the demo large-sized brick shells. The size of the bricks was 50 cm x 10 cm x 4 cm and the size of the foam glass blocks 50 cm x 10 cm x 45 cm. The bricks were first glued using a cement-based mineral glue to the foam glass blocks. Then the glue was applied on the clay brick shells to connect adjacent CTB blocks into a mechanically stable wall.



Figure 12: CleanTechBlock wall element mounted on insulation frame for Guarded Hot Box test.

The foam glass block orientation also influenced the tests performed on the CTB test wall In the test wall the orientation of the heat flow was parallel to the foaming plane thus the expected effective thermal conductivity was 47 mW/(m·K). This value is in accordance with the GHP test on the demonstration 1:1-sized sample giving a thermal conductivity of 48 mW/(m·K); the sample was measured in the in-plane direction. The Hot Box value for the CTB wall with foam glass oriented in-plane gave values of 62–70 mW/(m·K) @ 27–29 °C. The lowest value is equivalent to a value of 53 mW/(m·K) at 10 °C. The additional increase in the thermal conductivity for the wall tested in a Hot Box can related to imperfect junctions between adjacent CTBs as well as the different heat flow direction in comparison to GHP apparatus. Similar increase was observed also in a test wall prepared with commercial foam glass. The thermal conductivity of the bricks was measured to be 0.576 W/m·K using GHP apparatus. The results of mechanical testing in a previous project revealed that the CTB wall is five times stronger than the traditional cavity wall.



Figure 13: Thermal conductivity of the CTB foam glass: measurements with Guarded Hot Plate Apparatus EP500 and Guarded Hot Box setup.

<u>Task 2.3. Real size house.</u> We faced some problems with construction of the exterior wall of the house. The problems were solved and construction of the house will be completed in June 2019. CTB has also achieved European Technical Assessment ETA-18/0970 or EAD 170012-00-0404. CTB Building blocks for construction of external facades, installed with tile adhesive type C2T has also been CE approved.



Figure 14: Construction of a CTB demonstration house.

<u>Task 2.4. Customer response.</u> We had two workshops in both Aarhus and Copenhagen together with architects and engineers. The architects and engineers have given a very positive response to the CleanTechBlock project. We look forward to being able to show the house to even more potential customers in the fall. We have already received more inquiries for the big project. But we must have just finished building the house before we can do more marketing. The response of the potential customers will be collected after the demo house will be finished.

Task 2.5. Business plan. The business plan was updated in 2018 with the input of EU market and competitor analysis. At present, the project has not resulted in increased turnover, export and employment. But is has a high potential to lead to the above-mentioned targets. CTB has also achieved European Technical Assessment ETA-18/0970 or EAD 170012-00-0404 on 2018/12/13. CTB Building blocks for construction of external facades, installed with tile adhesive type C2T has also been CE approved. We plan to go to the market according to updated Business plan schedule.



Figure 15: Green building materials market and trend.



Figure 16: Sustainability and environmental benefits from CTB when comparing with competitors.

Dissemination of project results

The project results were disseminated through different means:

- Papers in scientific journals (6 papers published, 2 papers in review process, 3 papers in preparation)
- Presentations at scientific conferences (7 scientific conferences attained, 3 invited talks, 8 oral presentations, 2 poster presentations)

- Presentations to the students at Aalborg University (foam glass was presented to 4 generations of students, 8 projects on foam glass were conducted by the students)
- Construction fairs (3 fairs in Denmark and Germany)
- Through the demo full-sized house that will be finished in June 2019 (two workshops were organized for architects and engineers, after the house will be finished the stake-holders and customers will have chance to see the CTB solution and their response will be collected)

1.6 Utilization of project results

Research organizations, Aalborg University and Jozef Stefan Institute, exploit the results and research related to foam glass for teaching. A PhD student was part of the project and another PhD student started research on foam glass in October 2018 at Jozef Stefan Institute. New information and knowledge gained by the researchers will be utilized for preparation of follow-up projects. One project was granted by the M-era.Net agency in 2017 and the results are upgrading the results of this project financed by EUDP as well as increasing the potential of industrial exploitation of the CTB. Besides the scientifically relevant projects, researchers and Gråsten Teglværk discuss possibilities for collaboration with companies designing foam glass production lines as well as the companies producing foam glass.

The background knowledge of this project that exhibits commercial potential is protected by two patents. First one is related to CTB structure (granted in 2013) and second to the recipe for foam glass with improved insulation properties produced directly from waste glass (granted in 2018). The research of the present project on foam glass is in great part protected by the second patent. The new knowledge gained within this project is at present protected as a trade secret. This knowledge can be, depending on the need, patented or exploited for commercial production at a later stage based on the agreement between the parties. In the future Gråsten Teglværk would like to work together with a partner making foam glass (ex. Glapor) by the newly developed and patented recipe. When the demo house is finished, we will invite potential customers to see the first DGNB-certified single-family house in Denmark (German Sustainable Building Council – Deutsche Gesellschaft für Nachhaltiges Bauen e.V.). The business plan from 2018 will be further updated with the future customers' goals. The CTB product meet the expectations of the markets' demand for recycled products.

The energy policy objectives will be realized through the improved insulation and sustainability of the foam glass production as well as a decreased use of clay bricks in the wall structure. Thus the project contributes in a large extent to realizing the energy policy objectives.

The results have not been transferred to other institutions after project completion. A Ph.D. project is part of the project. Some of the results were presented to the students in Chemistry and Chemical Engineering at Aalborg University when we introduce the relevant semesters (e.g. 4th and 7th semester). According to these presentations, the students of these semesters will form groups and choose the projects that they like the most. The papers of the Ph.D. student and other relevant materials were distributed to the students who carried out the projects related to glass foams. Some of the results were presented at both external and internal conferences and meetings.

The PhD student has (co-)supervised four student project groups during his PhD. The student groups characterized commercial glass foams, produced glass foams based on research results to investigate the effect of either composition or storage (water uptake), and characterized their own glass foams, e.g., thermal conductivity. The PhD student presented his research results at three different conferences with five presentations (two posters and three oral talks) – SGT100 and ESG2016 in Sheffield, UK; PACRIM and GOMD2018 in Waikoloa, Hawaii; and PNCS and ESG2018 in Saint Malo, France. Furthermore, a sixth presentation will be given at ICG and GOMD2019 in Boston, US in June 2019. Except for conference presentations, the project and research results have been presented at different PhD courses as well as during an international stay at Research Complex at Harwell, Oxfordshire, UK. The candidate will defend his PhD thesis on 5th April 2019.

1.7 Project conclusion and perspective

The project revealed the background of the foaming process, which enables preparation of foam glass with improved insulation properties directly from waste glass, i.e. without the need for a re-melting step. Our research was focused on gaining new knowledge on the foaming mechanism, which is a complex process of several chemical reactions and physical processes. Furthermore, we assessed the heat transfer in the foam glass. Our results demonstrate how the thermal conductivity is influenced by the solid composition, gas composition and open-cell content. Using this knowledge we were able to prepare foam glass with thermal conductivity as low as 37 mW/(m·K). Our work beyond the state of the art also revealed that the theoretical low limit of the thermal conductivity is even lower. We anticipate that future work in follow-up projects will produce foam glass with even better properties.

In the project we also demonstrated the up-scaling feasibility for the developed foam glass recipe, which is of great importance for the commercial exploitation. In this regard, the partners are in contact with relevant companies working in the field of foam glass. We predict that a close collaboration with these companies will enable upgrading of the developed foam glass recipe to a pilot production and later to a full-industrial production. Furthermore, the demo CTB wall was characterized for the insulation performance and the demo full-sized house will demonstrate the fast construction and its advantages in comparison to conventional cavity wall.

The demo CTB house will be used for dissemination of the project to the stockholders, i.e. architects, customers, constructors, policymakers, as well as foam glass industry. The detailed market analysis and the updated business plan guarantee the success of the CTB concept. We anticipate that commercial exploitation will generate increased turnover, export and employment. Moreover, due to improved sustainability of the foam glass production as well as the CTB wall concept, a large amount of greenhouse gas emissions will be avoided.

Annex 1 List of publications

Annex 2 Thermal conductivity measurement of cellular glass samples (Department of Civil Engineering, Aalborg University)

Annex 3 Market and competitor analysis (Gråsten Teglværk)

Annex 1

Scientific papers in Peer-refereed International Journals

- R.R. Petersen, J. König, Y.Z. Yue, Evaluation of Foaming Behavior of Glass Melts by High-Temperature Microscopy, International Journal of Applied Glass Science, 7 [4] 524–531 (2016) DOI: 10.1111/jag.12185
- M.B. Østergaard, R.R. Petersen, J. König, H. Johra, Y.Z. Yue, Influence of foaming agents on solid thermal conductivity of foam glasses prepared from CRT panel glass, Journal of Non-Crystalline Solids, 465 (2017) 59–64. DOI: j.jnoncrysol.2017.03.035
- R.R. Petersen, J. König, Y.Z. Yue, The viscosity window of the silicate glass foam production Journal of Non-Crystalline Solids, 456 (2017) 49–54. DOI: j.jnoncrysol.2016.10.041
- M.B. Østergaard, R.R. Petersen, J. König, M. Bockowski, Y.Z. Yue, Foam glass obtained through high-pressure sintering, J Am Ceram Soc. 2018;101:3917–3923. DOI: 10.1111/jace.15574
- M.B. Østergaard, R.R. Petersen, J. König, Y.Z. Yue, Effect of alkali phosphate content on foaming of CRT panel glass using Mn3O4 and carbon as foaming agents, Journal of Non-Crystalline Solids 482 (2018) 217–222. DOI: 10.1016/j.jnoncrysol.2017.12.041
- M.B. Østergaard, R.R. Petersen, J. König, M. Bockowski, Y.Z. Yue, Impact of gas composition on thermal conductivity of glass foams prepared via high-pressure sintering, Journal of Non-Crystalline Solids: X 1 (2019) 100014. DOI: 10.1016/j.nocx.2019.100014
- 7. M.B. Østergaard, B. Cai, R.R. Petersen, J. König, P.D. Lee, Y.Z. Yue, Impact of pore structure on thermal conductivity of glass foams, in review process, Materials Letters
- 8. J. König, V. Nemanič, M. Žumer, R.R Petersen, M.B. Østergaard, Y.Z. Yue, D. Suvorov, Evaluation of the contributions to the effective thermal conductivity of an openporous-type foamed glass, in review process, Construction and Building Materials
- 9. R.R. Petersen, J. König, N. Iversen, M.B. Østergaard, Y.Z. Yue, The foaming mechanism of glass foams prepared from Mn3O4, carbon and CRT panel glass, in preparation
- 10. J. König, R.R. Petersen, N. Iversen, Y.Z. Yue, Application of foaming agent oxidizing agent couples to foamed-glass formation, in preparation
- J. König, D. Fabijan, A. Lopez, P. Cimavilla, M. A. Rodrigues-Perez, R.R. Petersen, Y.Z. Yue, M. Spreitzer, Comparison of open- and closed-porous foamed glasses, in preparation

Conference Contributions

- 12. R.R. Petersen, J. König, and Y.Z. Yue, Production of lightweight foam glass (invited talk). Workshop on gases and bubbles in molten glasses: from chemical engineering to geosciences, Paris, France.
- R.R. Petersen, J. König, M.B. Østergaard, Y.Z. Yue, Thermal conductivity of glass foams (invited talk), 25th International Congress on Glass, ICG, Boston, USA June 9-14 2019.
- 14. R.R. Petersen, Mechanism of Foaming Light-Weight Glass Foams (Invited talk), Cell-MAT 2018, Bad Staffelstein, Germany 2018.
- J. König, R.R. Petersen, Y.Z. Yue, Revealing foaming reaction during glass foaming using evolved gas analysis, 24th International Congress on Glass, ICG, congress, Shanghai, China 2016
- 16. J. König, R. R. Petersen, M. B. Østergaard, Y.Z. Yue, D. Suvorov, Foamed glass a sustainable load-bearing insulation material, ICG annual meeting, Yokohama 2018
- J. König, U. Hribar, A. Lopez-Gil, P. Cimavilla, M. A. Rodrigues-Perez, Rasmus R. Petersen, Yuanzheng Yue, Comparison of open- and closed-porous foamed glass, 25th International Congress on Glass, ICG, Boston, USA June 9-14 2019
- 18. J. König, R.R. Petersen, M.B. Østergaard, Y.Z. Yue, Crystallization in foamed glasses, ECERS congress, Torino, Italy 2019

- 19. M.B. Østergaard, R.R. Petersen, J. König, H. Johra, and Y.Z. Yue, 100th SGT and ESG 2016, Oral presentation, "Influence of foaming agents on both the structure and the thermal conductivity of silicate glasses", Sheffield, United Kingdom, 4-8th September, 2016
- 20. M.B. Østergaard, R.R. Petersen, J. König, M. Bockowski, and Y.Z. Yue, PACRIM 12 including GOMD 2017, Oral presentation, "Thermal conductivity of foam glasses prepared using high pressure sintering", Waikoloa, Hawaii, 21-26th May, 2017
- 21. M.B. Østergaard, R.R. Petersen, J. König, M. Bockowski, and Y.Z. Yue, PACRIM 12 including GOMD 2017, Poster presentation, "Foaming glass using high pressure sintering", Waikoloa, Hawaii, 21-26th May, 2017
- 22. M.B. Østergaard, B. Cai, R.R. Petersen, J. König, P.D. Lee, and Y.Z. Yue, PNCS ESG 2018, Oral presentation, "Effect of Macrostructure on Thermal Conductivity of Foam Glass", Saint Malo, France, 9-13th July, 2018
- 23. M.B. Østergaard, R.R. Petersen, J. König, and Y.Z. Yue, PNCS ESG 2018, Poster presentation, "Effect of Alkali Phosphate Content on Foaming of CRT Panel Glass Using Mn3O4 and Carbon as Foaming Agents", Saint Malo, France, 9-13th July, 2018
- 24. M.B. Østergaard, R.R. Petersen, J. König, and Y.Z. Yue, Effects of pore structure and gas phase on thermal conductivity of glass foams, 25th International Congress on Glass, ICG, Boston, USA June 9-14 2019.

Technical Reports

- H. Johra, Johra, H. (2019). Project CleanTechBlock 2: Thermal conductivity measurement of cellular glass samples, Department of Civil Engineering, Aalborg University. DCE Technical Reports, No. 263 <u>http://vbn.aau.dk/files/294602744/-Project Clean-TechBlock 2 Thermal conductivity measurement of cellular glass samples.pdf</u>
- 26. R.R. Petersen, Scaling Up Glass Foam Production, Department of Chemistry and Bioscience, Section of Chemistry, Aalborg University

Ph.D. Thesis

27. M.B. Østergaard (2019), Glass and Glass-Ceramic Foams produced by both Physical and Chemical Approaches. Ph.D. Thesis.