Final report 2017-12-14

1.1 Project details

Project title	Sustainable manufacture of brick and tiles with microwave energy
Project identification (program abbrev. and file)	64013-0538 MAGF
Name of the programme which has funded the project	EUDP (Det Energiteknologiske Udviklings- og Demonstrationsprogram)
Project managing company/ institution (name and address)	Danish Technological Institute (DTI) Kongsvang Allé 29 DK-8000 Aarhus C
Project partners	Aarhus University, Department of Geoscience The University of Nottingham, Microwave Process Engineering (MPE) group (formerly NCIMP) C-Tech Innovation Strojer Tegl Randers Tegl A/S Helligsoe Teglværk
CVR (central business register)	DK 5697 6116
Date for submission	2013-09-12

1.2 Short description of project objective and results

Hovedmålet var at halvere energiforbruget til brænding af tegl, samtidig med at energikilden transformeres fra fossilt brændsel til grøn energi (el). I dag anvendes over 90% naturgas til brænding af tegl på danske teglværker. Målet nås ved at modne en ny teknologi: Microwave Assisted Gas Firing (MAGF). Projektets forskningsindsats, herunder en PhD og en Post Doc, har skabt ny viden om mineralogi og dielektrisk opvarmning af keramiske materialer. En matematisk model for beregning af sammensatte materialers dielektriske materialer er testet og verificeret. Hos Teknologisk Institut er der etableret et MAGF pilotanlæg med kapacitet til at brænde 200 mursten. Succesfulde brændinger har vist, at det er muligt at anvende op til 44% elektrisk energi, mens der totalt opnås en energibesparelse på 25 %. Det reducerer gasforbruget med 50-55% i forhold til konventionel brænding, og samtidig opnås en reduktion af mens opvarmningstiden på omkring 40%. Energiforbrug, fossile brændsler og procestid er centrale problemstillinger i den danske teglindustri, og de dokumenterede forbedringer i pilotskala gør projektet til en succes.

The main objective was to decrease energy consumption by 50% for brick firing, while transforming the energy source from fossil fuel to green energy (electricity). Today, more than 90% natural gas is used for firing bricks in Danish brickworks. The goal is achieved by maturing a new technology: Microwave Assisted Gas Firing (MAGF). The project's research efforts, including a PhD and a Post Doc, have created new knowledge about mineralogy and dielectric heating of ceramic materials. A mathematical model for calculating the dielectric properties of a mix of clay materials has been tested and verified. At DTI, a MAGF pilot plant with the capacity to burn 200 bricks has been established. Successful firings have shown that it is possible to use up to 44% of electrical energy while achieving a total energy saving of 25% relative to conventional burning. This means a reduction in the gas consumption of 50 to 55% while reducing heating time by approximately 40%. Energy consumption, fossil fuels and process time are key issues in the Danish brick industry, and the documented pilot scale improvements make the project a success.

1.3 Executive summary

The main objective has been to halve the energy consumption for drying and firing of brick, while the energy source is transformed from fossil fuels to electricity, potentially from renewable energy. This can be obtained by using new MAGF (Microwave Assisted Gas Firing) technology.
Scientific research including a PhD and a Post Doc at the University of Nottingham has created the required knowledge of mineralogy and dielectric heating of ceramic materials to facilitate a secure and efficient conversion of traditional drying and firing technologies to MAGF.

The current source of power for drying and firing of bricks in Danish brickworks is over 90% natural gas. The process from raw materials to brick is optimized and managed in modern PLC controlled systems. But the individual "formulae" and drying and firing programs are largely based on experience, rendering it expensive and difficult to make even minor changes in the process and to develop new products.

The inspiration for this project was amongst others the results of an extensive pilot project conducted in England in the 1990's. Energy savings of up to 50% were obtained for a series of ceramic products in pilot scale kilns, using a combination of natural gas and microwaves firing (MAGF), and at the same time cutting both production time and fluoride emissions – both major issues in today's production. The pilot project did not take advantage of the advanced modelling possibilities available today and lacked control of the temperature when heating was based mostly on microwaves. Thus only 10% microwave energy was applied.

In the current project, the firing cycles have been scaled up and optimized, and we have managed to apply up to 44% microwave energy when firing standard bricks in the pilot kiln at DTI.

The main activities of the project were

- Scientific research in typical Danish raw materials, their dielectric properties and response to microwave energy, performed mainly at the University of Nottingham.
- Scientific research in the mineralogical development of both traditionally fired and MAGF products including establishing thermodynamic models, performed at Aarhus University and the University of Nottingham.

- Establishment of an experimental pilot plant for MAGF firing of brick at DTI, targeted at the research activities of this project, for testing the models and optimizing firing cycles for MAGF firing.
- Industrial research collaboration at the brickworks, drawing on the knowledge of raw materials and brick firing to optimize firing cycles and implement the results of scientific research.

The project's ultimate objective was to determine the dielectric properties of the raw materials used in the brickworks, to create a basis for introducing MAGF technology in the plants. The mineralogical, thermodynamic and di-electric properties have been developed, and tested in the pilot plant, so it is possible to predict the effect of the new firing technology on product quality and energy consumption.

The strong modelling capacities at the Microwave Process Engineering (MPE) group, formerly NCIMP, has established a model for calculating the dielectric properties of clay mixtures, and a model for the electric field distribution in a kiln. This will in the future facilitate the development of new raw material mixes as well as the possibilities for better temperature control and thus, much better kilns.

The ultimate goal for the project was that the MAGF technology could be considered mature and ready for implementation in the industry in general. This may not be entirely true – yet. But the project has developed the tools and documented the potential of the MAGF technology to save energy, shorten process time and enhance product quality.

1.4 Project objectives

The projects overall objective was to halve the energy consumption for drying and firing of brick, by transforming the energy source from fossil fuels to electricity, potentially from renewable energy. The means to achieve was to mature the new MAGF (Microwave Assisted Gas Firing) technology.

Why microwave processing?

During microwave processing, energy is directly and instantaneously deposited into the material through interaction of its molecules with the electromagnetic field. This "volumetric heating" means that microwave processes do not rely on conventional heat transfer, which is often dominated by thermal conductivity limitations; rather than gradual heat transfer from the outside to the inside of the material, volumetric and uniform heating of the whole load can occur instantaneously, thus allowing rapid heating of large and complex shaped loads. During firing and sintering, the resulting minimization of thermal gradients within the material, lower grain growth and higher densification often translates to an overall product quality increase compared with conventional treatments.

Microwaves also heat "selectively", which means that the different components within a heterogeneous material such as clay may heat at different rates. Some materials are impervious to microwaves, and simply reflect them. Others are essentially transparent to microwaves, allowing them to pass through the material without being absorbed. Materials that absorb microwaves are known as dielectrics. In order to understand the potential to process a given material using microwaves, and to design appropriate cavities in which to do this, it is necessary to understand the dielectric properties of the material. These are comprised by a dielectric constant (ε') which measures the capability of the material to absorb microwave energy, and a loss factor (ε'') which describes the ability of the material to transfer the absorbed electromagnetic energy into heat.

The two motivations for using microwaves in this project were to (a) reduce energy requirements of the Danish brickworks industry and (b) to enable the Danish brickworks industry to determine the potential to substitute fossil fuels (gas firing) with renewable fuels (electricity). Conventional heating rates are limited by the formation of thermal gradients within the load, which result in cracks in the product. Volumetric heating would reduce these thermal gradients, hence reducing the overall heating time. This would potentially result in energy savings as the heat losses would be less if the processing time could be reduced. Replacing a portion of the energy with electrical heating would also facilitate the reduction of fossil fuel consumption.

The objectives were obtained through scientific research, and by establishing a pilot MAGF kiln and performing series of experimental firings of clay brick.

The project's scientific research objectives consist of the following parts:

- To establish thermodynamic and reaction kinetic models that can predict the mineralogical development from clay to brick, including determining how the natural variations in Danish raw materials can be utilized to create the desired physical, chemical and aesthetic dynamic properties of building bricks (bricks and tiles).
- To gain accurate knowledge of how much and how the process and thermodynamics influenced by applying respectively dielectric and gas-based firing technologies, as well as variations in the firing atmosphere.
- To study the dielectric properties of Danish raw materials for brick, and based on these characteristics, to optimize the use of microwave energy for drying and firing.

The following hypotheses H1, H2 and H3 are investigated in the project:

H1: Variations in the clay size fraction has significant influence on the reactivity and availability of elements.

H2: For each raw material and their chemical composition it is possible to model mineralogical phase changes as a function of temperature and time.

H3: With the use of MAGF (microwave assisted gas firing) it is possible to achieve significant energy savings from drying and firing, a faster production, and better quality".

In this section, the implementation of the project is described through the four work packages that address these objectives. Under here the risk management and changes of the project plan are described, as well as the fulfilment of the milestones, mentioned under each work package.

Work package 1: Mineralogy, thermodynamics and reaction kinetic models

WP1 manager: Hans Dieter Zimmermann, Department for Geoscience, AU

The research goal was to develop models that predict the mineralogical development in a given burned clay as a function of raw material and firing technology, which uses a combination of microwave and gas firing.

The processes for firing brick were explored and characterized, and thermodynamic established, that can predict the mineralogical evolution from clay to brick. On this basis it can be determined, how the natural variations in Danish raw materials could be utilized to create the desired physical, chemical and aesthetic properties of clay bricks and tiles.

Milestones

- M1 Determine the chemical, mineralogical and geological conditions in the selected clay pits.
- M2 Perform a series of detailed experiments that will uncover all factors during burning and their influence on the products.
- M3 Develop thermodynamic and reaction kinetic models that can predict the mineralogical evolution from clay to brick.

M1 was delivered through WP1.1 Material characterization.

M2 was delivered through WP1.2 Laboratory-scale firing experiments.

M3 was delivered through WP1.3, though it was no possible to establish a reaction kinetic model, due to uncertainties in the thermodynamic models.

WP1.1 Material characterization

To address the mineralogical changes occurring during the firing process of clay to brick a detailed characterization of the starting material (that was provided by the 3 participating brickworks), as well as the fired products was performed.

A total of 30 samples were provided by the brickworks representing the various raw clays, clay mixes for yellow and red brick production along with burned bricks.

The detailed characterization of the materials was performed using grain size analysis, separation of clay fractions, Differential Thermal Analysis (DTA), Differential Thermal Gravimetry (DTG), X-ray diffraction with/without simultaneous heating (XRD) and including Rietveld refinement of bulk and clay fractions, X-ray fluorescence (XRF), and polarisation microscopy.

Chemical compositions determined by XRF were used for modelling the thermodynamic mineral reactions that occur during the firing process (WP1.2).

WP1.2 Laboratory-scale firing experiments

Two series of laboratory-scale firing experiments were performed.

Firstly, small (0,1 g powder) fractions of clay with different grain sizes, were heated using temperature controlled XRD. This allows the observation of mineral phase changes during the heating process. This lead to an interesting observation. It was found that the temperature at which calcite and kaolinite break down (release of CO_2 and OH) is depending on the particle size. Smaller grains generally reacted at lower temperatures. This was one of the hypotheses to be tested in the project.

Secondly, the firing process used at the brickworks was simulated at the laboratory-scale where smaller samples were fired and collected at different temperatures from 300-1060°C to document the mineralogical development with temperature (Fig. 1).

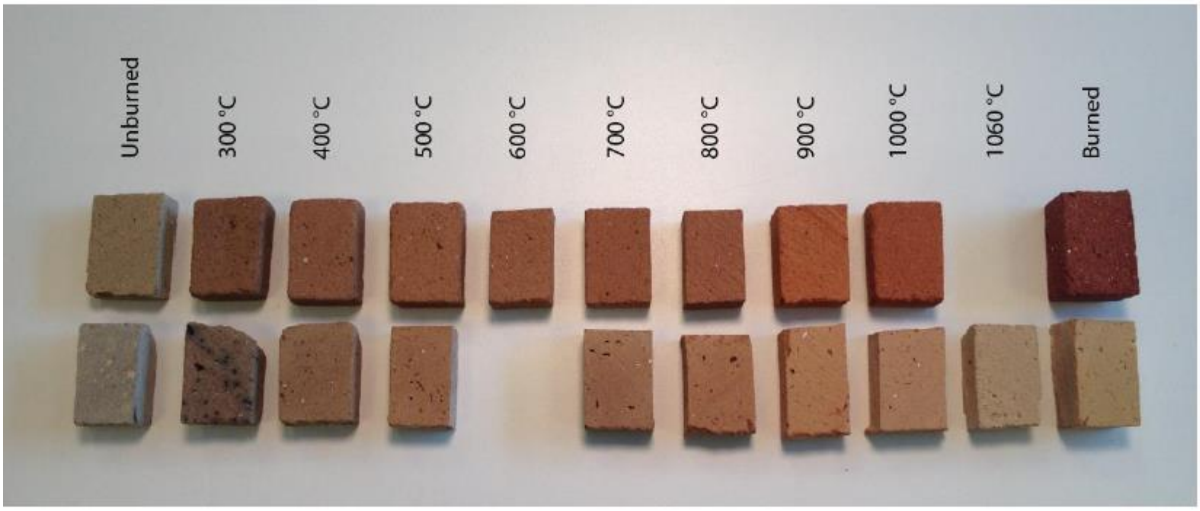

Fig. 1. Laboratory-scale 'bricks' showing the change in colour at different firing temperatures. Top row red-burning clay, lower row: yellow-burning clay.

The laboratory-fired samples, an unfired sample as well as a corresponding sample fired at the brickworks were crushed and analysed by XRD to determine their mineralogical compositions. This showed that during the firing process systematic changes of the sample mineralogy with increasing temperature occurs. The absolute amount of minerals in the samples was estimated using the peak areas, which were recalculated to percentages following the principles of Johns et al. (1954). Then the amount of minerals was normalized to 100%.

Change of modal composition of yellow and red firing bricks can be seen in Fig. 9 and 10 in section 1.5.

The bulk sample chemical composition was analysed by XRF and then compared with the Rietveld refinement analysis of the mineral phases from XRD analysis.

There is good agreement between the different approaches in particular if the chemical concentrations are larger than 10 weight%, which is typically the case for CaO (yellow bricks), SiO_2, and Al_2O_3. Quantitative phase analysis (QPA) by Rietveld analysis presumes that the sum of all weight fractions is equal to 1. Presence of undetected material invalidates this assumption, which can lead to some uncertainty in the calculation of the mineral phases and their composition.

Comparing the estimates from the Rietveld refinement with the modal estimates from peak width gives some differences in mineral phases that contain Na, Ca, K. These are mainly the feldspars and they often form solid-solutions. If the endmembers are used this can lead to an overestimate or underestimate of components. Similarly, clay minerals are chemically different and are treated as one group in the calculations. Equally, Rietveld refinement does not differentiate different clay types and the minerals calculated from the XRF data are supposed to be in equilibrium, which might not be the case in the samples investigated. Moreover, it was suspected that possibly some amorphous (not crystalline) phases have formed due to melting of the material (see below). This would not be possible to detect by XRD and will lead to an overestimation of the crystalline phases in the calculation of the amount of minerals. This could be a problem because mineral phases from the XRD are normalized to 100%.

To further investigate whether amorphous material influenced the modal estimates, several samples of the laboratory-scale firing experiments were cut to thin sections and investigated with a polarization microscope. The aim was to check the different textures at different temperatures and to see if any amorphous phases (i.e., melting of material) in the bricks have formed. The following observations were made: (1) the bulk – i.e. up to 95 vol% – of each sample is made up of a crypto- to microcrystalline matrix and of sand-sized quartz grains. (2) Besides quartz, plagioclase, microcline and hematite are present in all samples (Fig. 2). This is in agreement with XRD results. There is no indication of major melt formation. To further assess the presence of potential amorphous phases quantitative XRD analyses of selected samples were made. No significant amount (<5%) of amorphous material was detected.

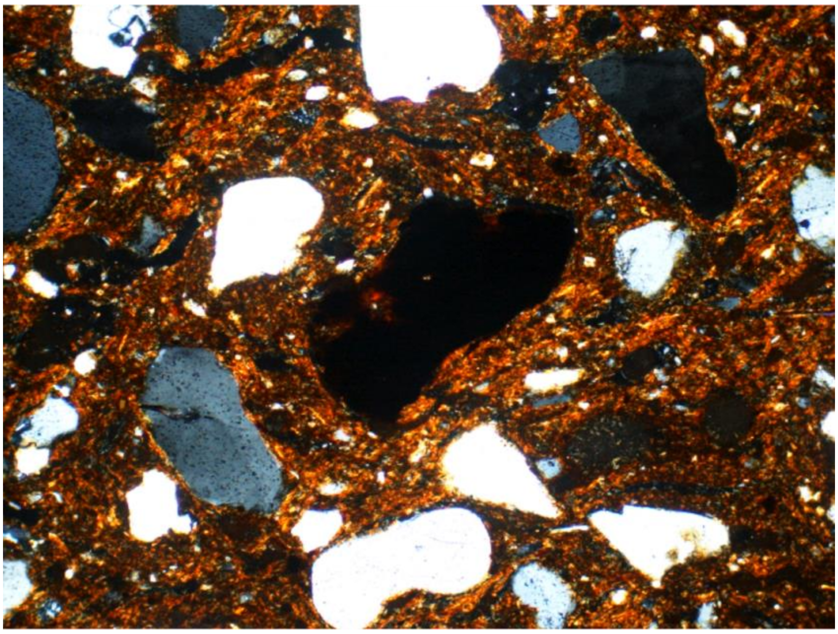

Fig. 2. Thin section photograph of red clay sample fired at 900°C. Grey and white grains are quartz, central dark phase is opaque. The reddish brown colour of the matrix is most likely due to hematite. Width of photo 2 mm.

WP1.3 Thermodynamic modelling

In addition to the modal composition, the bulk chemical compositions of the materials were determined by XRF. This data was used to model the equilibrium phase assemblage for temperatures up to 1250 °C using the Gibbs free minimization algorithms in the Theriak-Domino software and data from various thermodynamic databases.

The model calculates the most stable mineral assemblage under the given conditions, and hence the phases that will ideally form at the given temperatures. Fig. 3 shows the calculated equilibrium phases for the yellow burning clay from Hammershoj brickwork (sample “Hammershoj Yellow”).

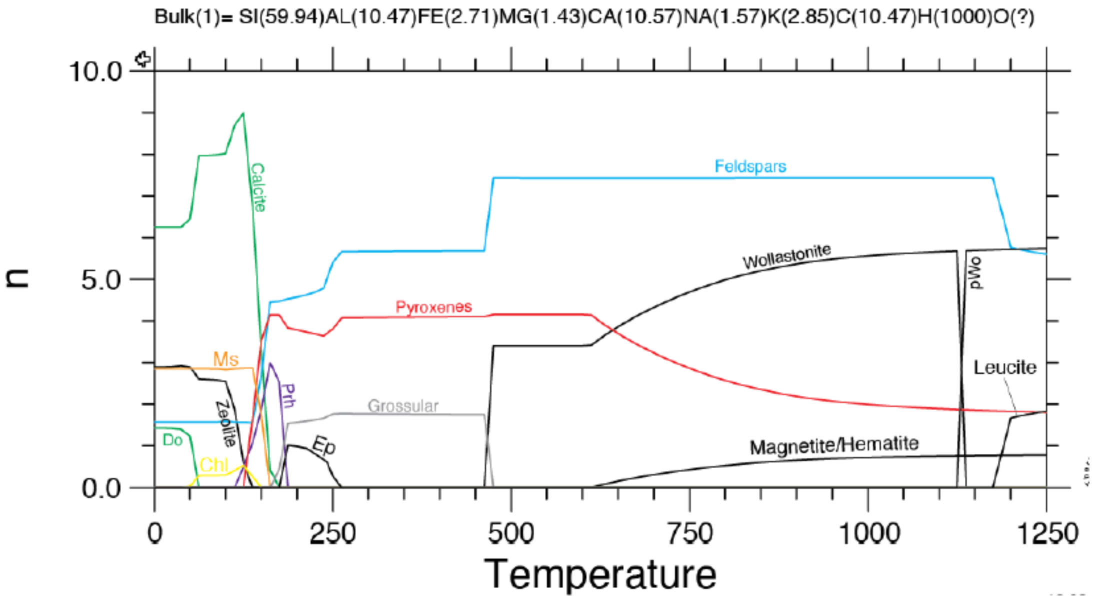

Fig. 3. Theriak-Domino phase diagram based on yellow burning clay from Hammershoj brickwork.

The mineral assemblages predicted by the Theriak-Domino phase diagrams were then compared with the modal composition of the fired laboratory samples. The phase diagram shows the minerals, which are most likely to form if reactions occur, and hence, is of little relevance at low temperature until the clay minerals decompose, and formation of new mineral phases begins (around 500°C).

At higher temperature there was good consistency between the mineral assemblage in the model and experiments (mainly quartz, feldspars, pyroxene, wollastonite and hematite). The main difference in the mineral assemblages predicted for yellow and red brick, is that following the breakdown of calcite, the higher Calcium content in the yellow brick means that a higher fraction of the iron is bound in ferric calcium silicates (pyroxene and wollastonite) instead of iron oxides. This corresponds well with the phase assemblages in the experiments and probably explains the colour difference between these brick types.

However, the thermodynamic models also had shortcomings and predicted some minerals that were not observed from the experiments (cordierite and olivine), and mineral assemblages containing minerals that cannot coexist in equilibrium (leucite and quartz). Finally attempts to add at liquid phase (melt) to the model resulted in predictions of sudden complete melting at around 900°C, whereas we had difficulties detecting any traces of melt in the laboratory samples even at higher temperatures. These inconsistencies are likely signs that some of the thermodynamic data in the database have been extrapolated outside of the composition, temperature and pressure space that they were determined for

Finally, challenges were met in comparing the modelling and laboratory results quantitatively. Firstly, the phase diagrams from Theriak-Domino gives modal compositions in mole fraction while the mineral analysis results are in weight%. The conversion is not straightforward because several of the minerals are composed by a solid-solution between several end-members.

It was originally the plan to establish a reaction kinetic model, and with the described indications of the importance of grain size and reaction time for mineral conversions, it would also be extremely attractive. However, the reaction kinetic model had to be abandoned due to the many uncertainties regarding amorphous materials and the high complexity link between indications of amounts in the phase diagrams and in the mineral analyses. The complexity was estimated to be too high in relation to the yield that could then be achieved by theoretical path versus relatively simple trials.

Summary WP1

The research in this work package has fulfilled the proposed objectives and has documented and characterized the materials used by the brickworks. Furthermore, the work showed the mineralogical differences in the starting materials and their effect on the final mineralogy of the fired bricks.

The different analytical approaches as well as the thermodynamic calculations agree broadly and most of the expected mineral phases at different temperatures were also found in the samples. Nevertheless, the absolute amount of mineral phases differs somewhat from the different calculation methods. This is the main reason for abandoning the reaction kinetic modelling.

The results of the material characterization were used in WP2 to test the dielectric properties of the different mineral phases and to model the interaction with microwaves (see below).

Work package 2: Scientific research: Dielectric properties of yellow and red burning clays and microwave firing experiments

WP manager: Eleanor Binner, Assistant Professor, MPE, University of Nottingham

Partners: MPE with assistance of all

The main goal was the characterization of how red and yellow clays heat in a microwave field through knowledge of their dielectric properties in order to assess the potential for microwave processing. The factors that determine the dielectric response of clays were investigated and used to establish a predictive model capable of estimating the dielectric properties of any natural or synthetic clay mixture based on its composition. Ultimately, knowledge gained form the dielectric property characterization was used to design a laboratory scale microwave applicator capable of firing clay specimens without the need for conventional heating or the addition of microwave susceptors. The product quality of the microwave-fired specimens compared favourably with those fired using conventional heating.

Milestones

- M4 Performing a series of detailed small scale experiments that will uncover all factors during drying and firing with dielectric heating, and their influence on tile product
- M5 Determining the optimum proportion of microwave energy to the specific raw materials

M4 was delivered by MPE through three activities (WP2.1-2.3). The mineralogical assessment carried out by AU for WP1 was used as an input into WP2.2.

M5 was achieved with MAGF optimisation experiments carried out by DTI. The electromagnetic model of the MAGF furnace (MPE) WP2.4, based on data from M4, was used to understand and optimise the operation of the MAGF pilot kiln.

WP2.1 Measurement of the dielectric properties of red and yellow Danish clays and their major constituents at different conditions of temperature, microwave frequency and density.

WP2.2 Development of a predictive tool capable of estimating the dielectric properties of a clay mixture from its composition using electromagnetic mixing rules.

WP2.3 Performance of laboratory scale microwave heating trials on red and yellow clays on heating cycles akin to those used in brickworks and assessment of product quality compared with conventional products.

WP2.4 Electromagnetic simulations. WP2.4 was not part of the project plan and substitutes detailed investigation of drying.

WP2.1 Dielectric property characterization of clays and clay constituents

Two sets of raw yellow and red clays provided by the participating brickworks were chosen for the dielectric property study. Moreover, the major clay constituents were also analysed, i.e. kaolinite, montmorillonite, muscovite, quartz, calcite, K-feldspar and plagioclase. Dielectric properties of clays and clay constituents were measured in triplicate at frequencies close to the industrial ISM microwave frequencies of 912MHz and 2470MHz by using the cavity perturbation method. Measurements were performed from room temperature up to 950ºC on the specimens in the form of crushed powders and carved chunks.

WP2.2 Development of dielectric properties predictive tool

Dielectric property measurements are time consuming, and can only be carried out in specialised microwave metrology laboratories. In order to provide an understanding of how any clay material may heat during microwave processing without the need for dielectric property measurements, a tool capable of producing fast and acceptable predictions was sought. A set of electromagnetic mixing rules was selected in order to attempt the estimation of the dielectric properties of any clay mixture using the dielectric properties of the constituents measured above and the unfired clay composition. The mixing rules used are Rayleigh, Sen-Scala-Cohen, Böttcher, Complex Refractive Index, Landau-Lifshitz-Looyenga, Lichtenecker and Bruggeman-Hanai. Water is not considered a major clay constituent in the calculations given its disappearance well before firing temperatures are reached. Instead a correction to the loss factor was added through cross multiplication between moisture contents. Validation of the estimations was done by comparison with the dielectric properties of red clays from England and the Netherlands, yellow clay from Spain and the Danish clays above. The study at different frequencies, densities and with variable compositions allows for the consideration of the application limits of the predictive tool.

WP2.3 Performance of laboratory scale microwave heating trials on red and yellow clays on heating cycles akin to those used in brickworks and assessment of product quality compared with conventional products.

Laboratory scale trials were performed using a WR-430 single mode applicator working at 2450MHz. In order to reach thermal uniformity on heating and due to cavity restrictions, the samples were extruded cylinders of 25x20mm. Initially, heating trials at constant input powers ranging 100W-300W for 80min served to investigate the affinity of the dielectric property measurements to the experimental performance of the clay under microwave energy, assessing any possible deviation resulting from intense reactions among the constituents. Afterwards, a heating profile reminiscent of that used in brickworks is applied by controlling the input power. In doing so, first the temperature of the clay and later its energy needs are established as the dependent variables. Both being monitored by thermal camera and power meter measurements respectively. The heating cycles last around 150min with 30min of holding time at a top temperature averaging at 1050˚C and with natural air convection cooling.

Given the size and shape differences of the fired specimens when compared with industrial bricks, the resultant quality comparison would be flawed. Thus, raw specimens of the same dimensions and geometry were conventionally fired at a typical brickworks cycle of 55˚C/h up to 1050˚C, 4h soaking and 20˚C/h cooling down to room temperature. Quality comparison is focused on the compressive strength, 24h cold and 5h hot water absorption, saturation coefficient and colour change.

WP2.4 Electromagnetic simulation

Using the data from M4, an electromagnetic model of the MAGF furnace (MPE) was built. In conjunction with MAGF optimisation experiments carried out by DTI, this model was used to understand and optimise the operation of the MAGF.

The simulation work was underpinned by building a real model of the MAGF and measuring the dielectric properties of the bricks at different temperatures from 20°C up to 1000°C to mimic the dielectric properties during firing. Simulation were carried out using COMSOL Multiphysics®, a finite element analysis simulation software. It allows the calculation of physical parameters such as electric field and power density, which are vital to establish the uniformity and efficiency of the microwave treatment. The work aimed to establish the effect of brick position inside the kiln and advise on the load size and distribution required to maximize the efficiency of the system and reduce energy consumption. Fig. 4 represents a 3D model of the MAGF presented in COMSOL; it shows the microwave input (4 possible microwave feeds) and the internal dimensions of the kiln.

Initially, the model was built using the dielectric property measurements from WP2.1. The results from this model were presented to the partners at a project meeting in January 2017 in order to aid DTI in the planning of the MAGF experiments. However, a different dielectric property measurement technique was required to validate the dielectric properties of the whole bricks. This “waveguide measurement” technique required the design of a bespoke apparatus that allowed measurement of half bricks up to firing temperatures; this apparatus was not completed until November

2017, and is therefore not included in this project. Unfortunately, there wasn't time for the fully updated electromagnetic model to inform further optimization experiments. This was in part due to the limited experiments that were possible in the MAGF due to fabrication delays, and in part due to the requirement to fabricate a new dielectric property measurement technique to validate the model.

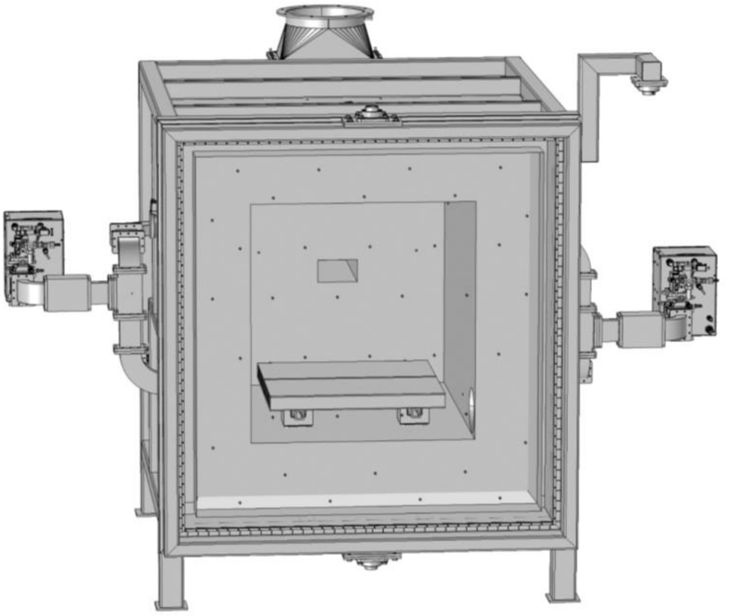

Fig 4: 3D model of the MAGF imported into COMSOL Multiphysics®.

Summary WP 2

The research in this work package has fulfilled the proposed objectives.

M4: The dielectric properties of clays used in brickworks and their major constituents have been studied across the firing range and their influence on the microwave process ability of the overall clay was assessed. The single minerals data was successfully used in developing a dielectric properties predicting tool in order to avoid the measurement of an infinite number of clay mixtures. The results of this study were used in the design of microwave heating trials that deepened the understanding of the microwave/material interaction and allowed the comparison between conventional and microwaved clay products.

Note that, based on a discussion with the brickworks' partners early in the project, the initial aim to investigate microwave drying as well as firing was abandoned, as in the brickworks, waste heat generated during firing is used to dry the bricks at no additional energy cost. The use of electricity instead of waste heat integration therefore wasn't deemed appropriate.

M5: The optimisation of the MAGF furnace with support from the electromagnetic model has provided a good insight into the optimal operation of the MAGF furnace and hence the optimal proportion of microwave energy that should be applied. Through experimental firings in the MAGF furnace it has been proven that 50% of energy for firing can be supplied from microwaves. This is limited only by the capacity of the microwave generators of the furnace.

Work package 3: Establishing and running a pilot plant at DTI

Project manager: Abelone Køster, DTI

The main goal in WP3 was to develop and establish a MAGF pilot plant (kiln) with microwave drying and firing of brick products. This would include the first experiments and scale-up of the results of WP1 and WP2, as well as investigating energy consumption and product quality. The theoretical models are tested and adjusted. Experiments performed in parallel at the brickworks allowed the comparison between MAGF fired products and conventionally fired products.

Milestones

- M6 Pilot plant for drying gas and microwaves is established. Pilot plant for MAGF firing is established.
- M7 First experiments are conducted
- M8 Testing the models from WP1.

In the original project plan, it was quite optimistically planned, that the pilot plant should be in operation after 3 months from the project start. This soon turned out to be unrealistic, as it was difficult to find a supplier who would take on the task at all. This was due to the fact that the combination of gas heating and microwave heating is completely new in the industry, and it was very uncertain whether it would be possible to achieve a good result and handle safety issues. Several of the brick industry's known kiln suppliers were contacted, but only Instalat in the Netherlands had expertise and experience in building smaller kilns and was interested in taking on the task. Instalat also had good references at Danish brickworks and would be able to test the finished kiln at the Instalat workshop before delivery to DTI.

It was also the ambition that the plant should be able to test both microwave drying and firing. In the initial stages of the project, this was discussed thoroughly, and the drying function was discarded quite early. There were two main reasons for this:

1) Costs for establishing both drying and firing would mean that only a small plant could be afforded. There was a need for a pilot plant that could fire bricks in a normal format and in stacks that correspond to the dense stacking of bricks in the tunnel kilns, in order to demonstrate the advantages of uniform microwave heating.

2) When the bricks are fired and heated to more than 1000°C, there will still be ample heat for drying the bricks from the cooling of the bricks, and the technology and facilities of the works are highly adapted to this approach and utilization of energy.

Changing both firing and drying technology at once would mean a greater barrier to introducing the new technology. Instead, it is more realistic to implement MAGF technology in smaller stages, for example, by refurbishing an existing tunnel kin or building a smaller batch kiln for blue damping of bricks.

During the initial stages of the project, the properties of the raw materials were examined under the influence of microwaves with 2.45 GHz and 910 MHz respectively. Since both the microwave generators and the entire furnace design have to be adapted to the selected frequency range, the design of the kiln could not commence until it was decided which frequency would be used in this project. The initial studies showed that 910 MHz would probably be more effective for brick firing. However, due to restrictions in Danish legislation it was decided to use 2.45 GHz instead. The wavelength has importance of penetration depth, but for a smaller furnace where the microwave generators are placed on both sides, 2.45 GHz is fully applicable. At a later stage of the project, it was also shown with simulation (WP2.4 Electromagnetic simulation), that 2.45 GHz could be satisfactory even for a tunnel kiln, given the proper design.

During the first year of the project these preliminary investigations and explorations were made while at the same time negotiations with possible suppliers took place. Subsequently, Instalat worked with the project team, especially C-Tech and DTI, to outline the plant and, in particular, discuss the responsibilities in connection with the design and construction of the kiln. The final agreement was that Instalat would build the gas kiln according to specifications from C-Tech regarding safety and materials selection. Instalat also took responsibility for designing the control system. Among other things, special and quite expensive materials are required for the internal insulation of the kiln, as it must not be heated by microwaves. No point welding is allowed and special chokes and safety measures must be incorporated into the door and the chimney.

C-Tech was responsible for the commissioning of microwave parts and generators, the production of custom-designed waveguides, as well as the monitored building of the gas kiln. C-tech also undertook the mounting and connecting of microwave parts. Finally, in June 2016, the kiln could be tested at Instalat in the Netherlands, where both DTI and C-Tech participated.

Kiln function and safety of microwaves were tested by C-Tech. There was no measurable release of microwaves. In July 2016 the kiln was delivered to DTI in Aarhus and the first test runs were made immediately.

Fig. 5a: A forklift with a lifting capacity of 5 tonnes was used to transport the pilot plant from truck to final placement in a basement under the DTI.

Fig. 5b: First test run in Aarhus after delivery. The kiln is equipped with 4 gas burners and 2 microwave generators, each with 2 ports.
The power supplies for MW generators are water-cooled.

The brickworks supplied dried stones ready for firing. A number of initial firings were made where the primary purpose was to gain experience in controlling the kiln and achieving the desired temperatures in the goods. In particular, it was interesting to achieve a more uniform temperature distribution than known from conventional firing at the brickworks. There were a number of challenges in the management, especially cooling, which required minor adjustments of the plant and the control system.

M6 was met with delivery of the plant. The plant is not optimized for drying, but drying will in principle be possible with microwaves alone, as the ventilation can be controlled via the gas burner's air supply. This is not done in the project.

M7 is met with the initial test firings and commissioning of the plant. The capacity of the kiln was then optimized for firing 180 bricks of Danish normal format. Here it is possible to obtain the same conditions as in the models from WP1, i.e. **M8** is met. It was especially important to show that good results could be achieved with bricks in the same type of stacks as in the works, as the challenge is to achieve uniform temperature in a dense stack of bricks. Comparative firings have been carried out with gas alone and with MAGF, where both product quality and energy consumption and temperature profiles are analysed.

Summary WP3

A well-functioning pilot plant has been established which has met the requirement to demonstrate the potential of MAGF brick firing technology. The delay in establishing the plant, which became more than 18 months in relation to the original schedule, was reduced to 9 month’s total delay, as the project has been extended 9 months. This has not affected the basic performance of the project, but the ambition to hold an international conference has had to be abandoned, as the results have been so late in the project that there has been no time to plan one.

Work package 4: product development, controlling the physical, chemical and aesthetical characteristics of the fired clay

Project manager: Lars Peter Salmonsen, DTI

Partners: DTI and brickworks with assistance from universities and C-tech

Using the pilot plant for conducting experiments, models of the relationship between raw materials and process, and brick properties are studied through comparative testing. Bricks were fired in the pilot kiln using gas only and MAGF, and compared with bricks fired at the brickworks.

For the first series of test runs, simple evaluation criteria such as colour, cracking, "sound" and overall visual appearance were chosen. This enabled the project to move forward and test various firing cycles, quickly discarding non-successful firing cycles. Further testing with standard test methods were performed on bricks from a selection of the firing cycles, which were approved by the prior, simple evaluation methods. Testing with varied firing atmosphere were performed.

Milestones

- M9 Implement a wide range of comparative pilot and full scale production runs
- CM1 Brickworks individual plan for process improvement described.

M9 is met through 33 complete firing cycles since the pilot kiln was installed at DTI. Additionally, a series of shorter test firings were performed to investigate the kiln atmosphere at different settings, readjust the gas burners and test the influence of the microwaves on various temperature sensors. To enable comparison of the individual firings, all test runs were carried out on the same batch of dried clay bricks, consisting of red soft mud bricks and yellow burning ditto. Because of the delay of the project, and the need for comparison, mostly, the studies were carried out using red bricks. A series of test firings included some yellow-burning bricks, to gain experience with their qualities under the given firing conditions, but the firing cycles and optimization were targeted at the red bricks. In addition, it was necessary to use ballast in some firings, in the form of already fired bricks.

The first 11 test runs were included in the commissioning and adjustment of the kiln and consist of 4 runs with conventional gas firing and varying numbers of stones and stacking, to create a baseline compared with conventional brick firing. It quickly turned out that, using conventional gas firing, it is a challenge to heat all the bricks uniformly when the kiln is filled to its maximum capacity (198 stones). With this load, a longer dwell time (time at peak temperature) was required compared to tunnel kilns to ensure a uniform temperature throughout the stack. 7 test runs were performed to gradually increase the proportion of MW energy. These runs were with only 72 stones per firing, in order to save resources and time. It turned out that it is possible to fully utilize the two MW generators, i.e. to fire with 2x6 kW throughout the heating cycle, which is equivalent to around 40-45 % of the total energy consumption, depending on firing cycle and setting of bricks. This was more than expected.

There was some discoloration that could be indicative of runaway MW-heating or reduction. To test this, it was tried to induce stronger reduction by faster firing without aid of microwaves while adding organic matter (sawdust) that burned during the process. It could be concluded that the atmosphere had no influence. In the following run, it was found that with no MW power during the dwell time, discoloration does not develop. (2 runs).

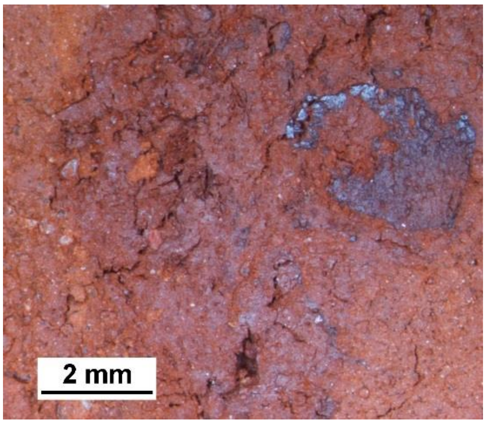

Fig. 6a. Surface discoloration in one of the first MAGF burns

Fig. 6b. Reduction core in the centre of stone fired with gas, with rapid heating.

Following this, a longer series of a total of 13 runs was performed, of which 9 with gas alone. The main target was to reduce the energy consumption of both conventional gas firings and MAGF runs by increasing the heating rate and reducing dwell time as much as possible. Simultaneously it was tested how much, and at which temperature, the microwave power needs to be reduced to avoid the aforementioned discoloration.

The main conclusion was that while MAGF-runs consume more energy than conventional gas firings with the same heating rate and dwell time, it is possible to save energy with the MAGF technique because the heating rate can be increased. This leads to a reduced process time, which in itself is very attractive for the brickworks.

Another important finding was that a bigger load and higher density setting is required to take full advantage of the benefits of the MAGF-technique. The reason is, that the relatively open setting allows uniform heating of the stack using conventional gas firing even at very high heating rates. Hence, there is not much room for increasing the heating rate using microwaves, before the process time is so short, that the time available for mineral reactions in the brick is insufficient.

Hence, the last 6 runs have been crucial for understanding the potential of the MAGF technology. Here, the number of bricks was increased to 180 placed in one stack, with a setting density corresponding to that in the brickworks. There has been a focus here on producing bricks with the same characteristics and quality as conventionally fired bricks. This is ensured by monitoring that the temperature in the stack is uniform during heating, whereby the dwell time can be reduced.

Test of bricks has been completed in order to compare the following 3 scenarios:

1. Bricks from the most successful firings with MAGF, i.e. as expected to have the same or better properties than conventionally fired bricks.
2. Bricks from conventional brickworks.
3. Bricks from the pilot kiln, fired with gas alone, but with the same heating speed and dwell time, obtained in the best MAGF burns (scenario 1).

Comparison between bricks of 1 and 2 must show that bricks fired with MAGF in the pilot kiln yield at least as good properties as conventionally fired bricks. Comparison between 1 and 3 must conversely show that when gas is the sole energy source used for firing in the pilot kiln, it is not possible to achieve shortening of process time and energy savings while maintaining product quality.

Fig. 7a: Early gas firing with dense load and resulting colour variations

Fig. 7b: Optimized MAGF firing, uniform colour

Comparison of bricks has been done visually, and partly with standardized methods as used by the brickworks to test and declare essential properties such as compressive strength, density, initial rate of water absorption and water absorption.

To fulfil **CM1**, for each of the brickworks participating in the project, an individual roadmap has been prepared, supplementing data for the work's own clay etc., and a suggestion for where MAGF is best implemented. An assessment of potential benefits of using MAGF, including increased plant productivity and potential energy savings and CO_2 savings through the introduction of green energy.

The goal is to judge whether it is worthwhile for the manufacturer to introduce MAGF for all or part of the production.

The individual plans are based on a very preliminary assessment, which assumes that MAGF technology is ripe for implementation. Thus, a real risk assessment of the extensive investment required cannot be made on the basis of this.

Summary of project development and changes

The project plan following the application, including milestones M1-M9 and CM1 was overall fulfilled. As explained above, there was a delay of about 18 months caused by the delay of design and commissioning of the pilot plant. This lead to an extension of 9 months of the project to end September 30th 2017. As explained above, drying with microwaves was abandoned due to the fact that the energy consumption for drying comes mainly from cooling bricks, and to avoid a substantial reduction of the pilot plant size for firing. Further the focus on yellow firing brick has been lessened with respects to experimental firing, again due to the delay, and extra work needed to adjust gas burners of the kiln – these turned out to be too powerful and thus difficult to control, not least because more MW power than expected could be applied. It was a specific goal to use as much MW power as possible, in order to be able to phase out fossil fuels.

The changes and extra challenges lead to a redistribution of funding in favour of DTI, UoN and C-Tech Innovation. To compensate for not researching drying, research in electromagnetic field distribution was performed (WP2.4) instead.

1.5 Project results and dissemination of results

1.5.1 Project results

The main challenges stated in the project proposal were:

- Lack of knowledge about the precise characteristics of clay and production process leading to the properties of the finished bricks.
- A high energy consumption, based on 90% fossil fuels in the firing process.

The project has overall succeeded in realizing its main objectives to solve the challenges:

Through research in dielectric heating and ceramic materials mineralogy, to create the foundations for a breakthrough in the Danish brick manufacturing. A knowledge base in the ceramic industry has been created, which is required to reschedule process energy from natural gas to electricity, thereby facilitating the use of renewable energy sources and achieve significant energy savings of 25% while maintaining product quality and increasing productivity through reduced process time.

The project's research components have provided ground-breaking new knowledge on the use of new green energy sources to one of the most energy-intensive industries in Denmark, as well as knowledge of how raw materials are mixed and utilized to achieve the desired properties of brick with a significantly lower energy consumption.

However, it was also the goal to mature the MAGF technology so that it could be implemented within the Danish brick industry within one year after the end of the project. We must acknowledge that this ultimate goal was too ambitious. There is still a need for further development and upscaling of the MAGF technology, before it can be commercially implemented in the brick and tile industry. It has been proven, on a pilot scale that bricks can be successfully fired with 44% MW power, replacing gas as energy source. But a further development of full scale production equipment is still needed.

The main project results are:

- A thorough mapping of Danish raw material and clay mixes for brick production. The materials have been characterized and analysed for mineral and chemical composition and grain size distribution
- Establishing a thermodynamic model for phase changes in during firing

- Mapping of the dielectric properties of clay mixes in the relevant temperature range for brick production (up to 950°C)
- Established and verified a mathematical model for calculation the dielectric properties for a clay mix, based on knowledge of the dielectric properties of each component
- Produced microwave-fired sample-size bricks without the assistance of conventional heating, which were of similar quality to conventionally fired samples.
- Developed a model using numerical simulation for estimating the electric field distribution in the MAGF pilot plant.
- Established a MAGF pilot plant at the DTI for pilot scale firing of bricks (up to 200 pcs standard bricks)
- Optimized the firing cycle for a relatively open setting (72 bricks), hence reducing total energy consumption by 7%, gas consumption by 41% and heating and dwell time by 25% compared to conventional gas firing.
- Documented, in pilot scale with dense setting comparable to brickworks (180 brick load), energy savings of up to 25%, while reducing gas consumption by 50-55% and decreasing combined heating and dwell time by 40% compared to conventional gas firing (although the firing cycles are not yet fully optimized)

The main activities have been described in more practical detail in section 1.4 relating to work packages and milestones from the project application.

The following description of main activities is with a focus on the results

Main results of the mineralogical research (WP1), AU

The main results are:

- Mapping of the clay minerals and grain size distribution of an extensive sampling of raw materials and production clay mixing from the brickwork partners. Examples shown below. The full data will be made available for the brickwork partners and for future research, in a separate report
- Knowledge of phase changes occurring under firing of red and yellow burning clays, based on the raw material, shown below in Fig. 9 and 10.
- Confirmed hypothesis *H1: Variations in the clay size fraction has significant influence on the reactivity and availability of elements,* where it is shown that smaller grains generally reacted at lower temperatures. Specifically, it was found that the temperature at calcite and kaolinite break down (release of CO_2 and OH) is depending on the particle size.
- It is possible to use Theriak-Domino software to model and predict phase changes during firing, based on mapping of the clay minerals of the raw material. The model still needs some adjustment.

The results of the analyses of 30 samples provided by the brickworks showed (as expected) distinct difference between red and yellow clay starting materials (Fig. 8). A major difference is the calcite content of the yellow clay that needs to be considered during the firing since the breakdown of calcite and escape of CO_2 will cause changes in the bulk volume. This was observed in the DTA analysis (Fig. 8) of the raw clays. The samples were also chemically analysed by XRF and results were used for modelling the thermodynamic mineral reactions during the firing process (see below).

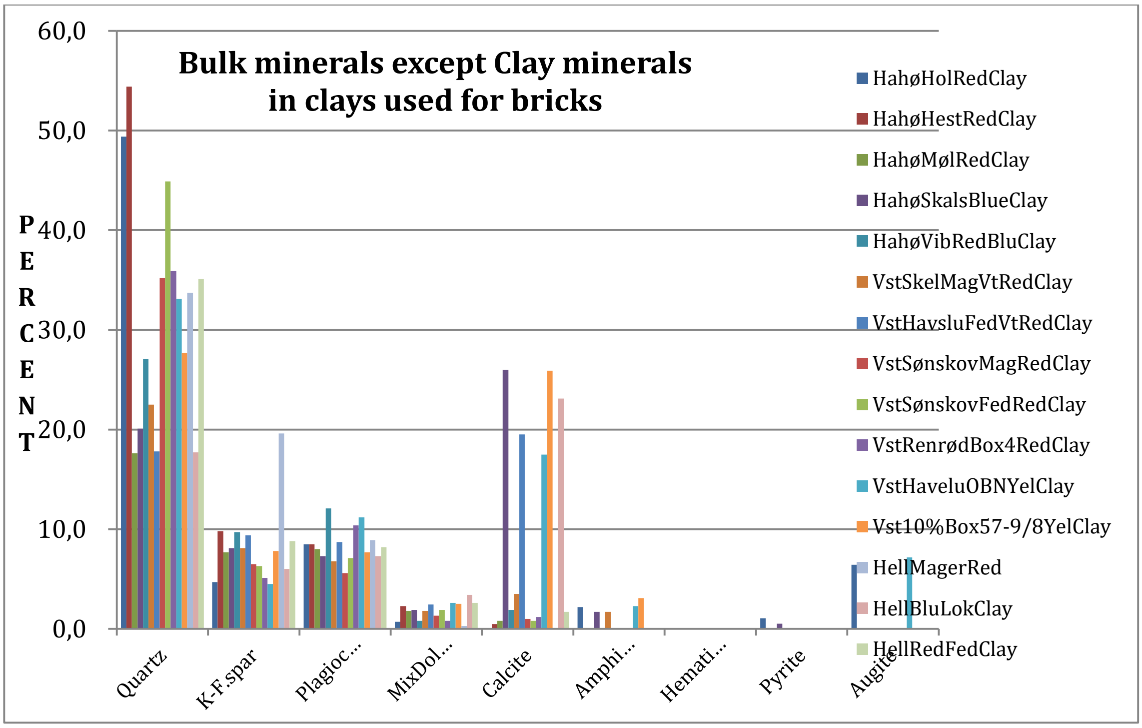

Fig. 8. Mineralogical composition of the investigated raw materials (different types of clay). Red and yellow clay show main differences in the calcite content.

Mineralogy of fired brick samples

A remarkable result is that Theriak-Domino predicted calcite decomposition starting around 200°C, which is very early compared to the experience from brickworks and cement plants where the breakdown is known to occur mainly around 800°C. Further models showed that in systems that only contain calcite, the breakdown of calcite to lime and CO_2 is predicted around 800°C and that this is only affected slightly by the water and CO_2-pressure. However, if silica is added to the system, calcite and silica reacts to form wollastonite (or pyroxene if iron is also present) already at 200°C. Although the exact temperature is uncertain, the result indicates that, if silica is available for the above reaction, calcite decomposition may start significantly earlier than previously anticipated.

The prediction is supported by the results of the temperature controlled XRD analysis, which surprised by showing that calcite breakdown had started at 475 °C in the finest grain fractions. This could indicate that reaction between the finest calcite particles and silica begins as soon as breakdown of clay minerals results in fine-grained reactive silica. And although this is the thermodynamically favoured reaction it is limited by slowness of solid state diffusion, and hence, the majority of the calcite does not breakdown until the decarbonation starts at 800°C.

In yellow bricks, a high amount of augite (45-50%) forms during firing. The higher augite content is related to the high content of calcium derived from the breakdown of the calcite in yellow-burning brick. The CO_2 from this reaction is released from the system, whereas calcium reacts with Fe and Si from the clay minerals to form augite. In some samples, the amount of quartz decreases from unburned to burnt samples because it is used in augite and to some extent in feldspar. In other samples, the quartz content is near constant because the release of Si from the breakdown of clay minerals is balanced by the formation of feldspar (Fig. 9, 10).

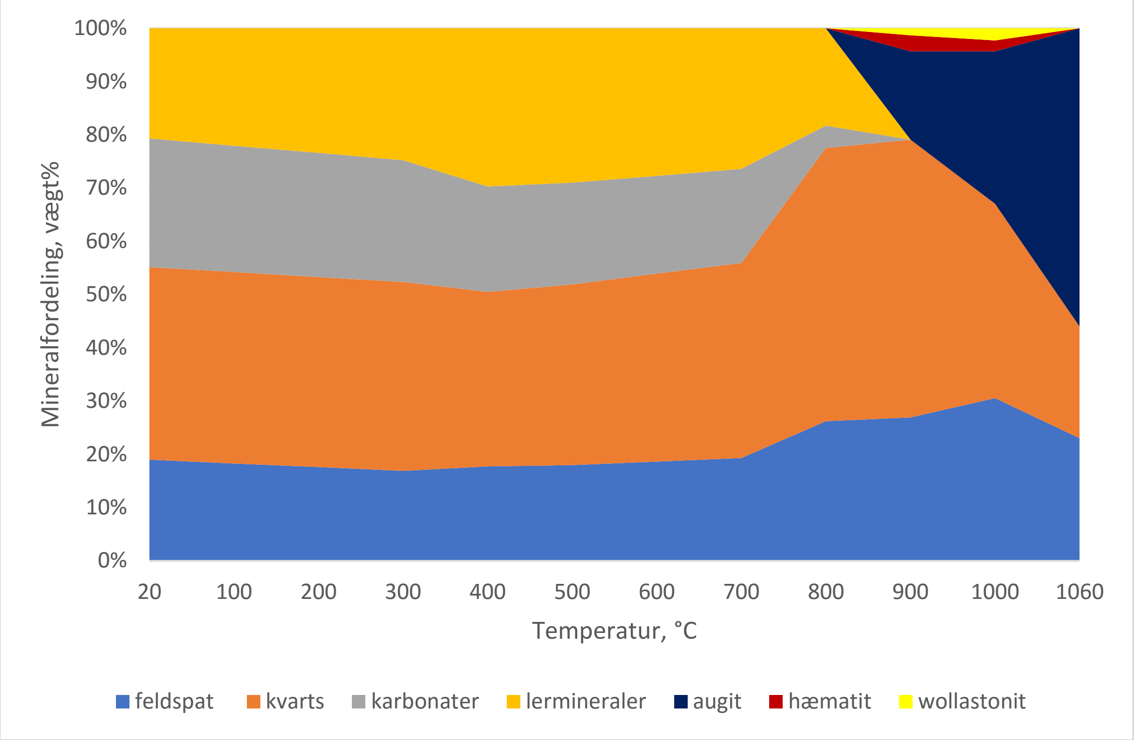

Fig. 9. Change of modal composition of yellow-fired brick from Vedstaarup at different temperatures. It can be seen augite is dominating in the fully-fired bricks

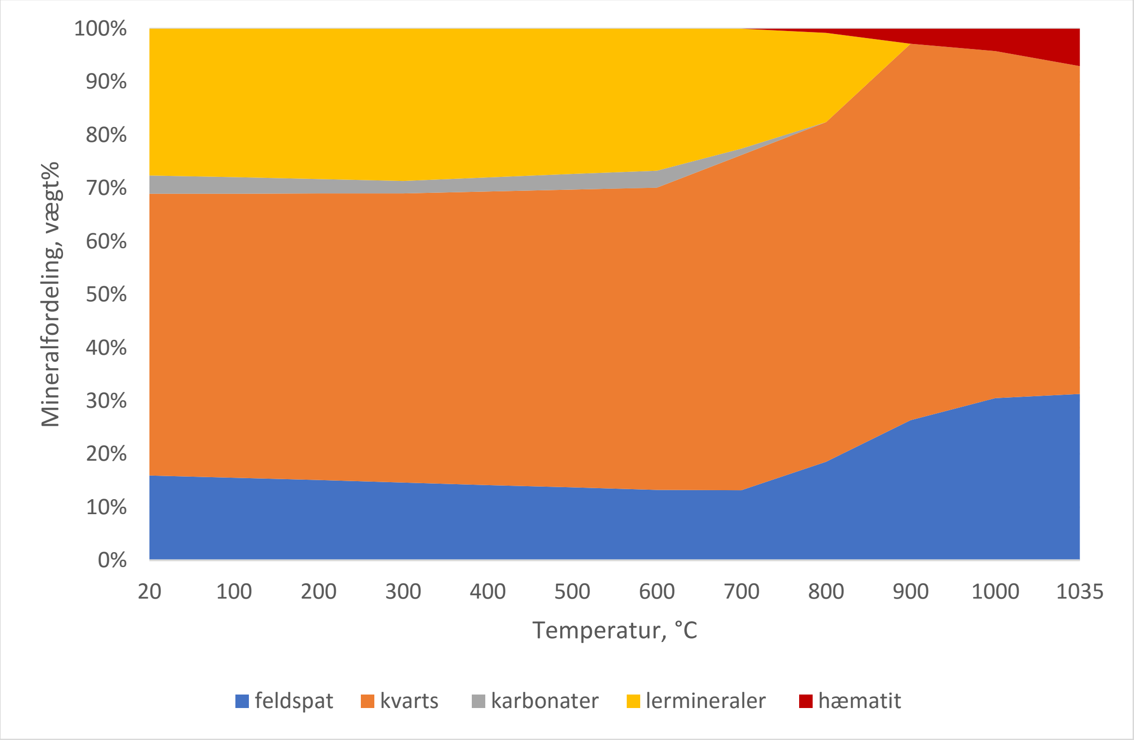

Fig. 10. *Change of modal composition of red-fired brick from Vedstaarup at different temperatures. The low calcite content in the starting material influences what the final modal composition of the fully-fired bricks are. There is no augite, but instead a higher amount of plagioclase and also hematite, which leads to the red colour of the brick.*

In red bricks it seems that there is no or little variation in the quartz content between the burned and unburned bricks. The destruction of clay minerals during heating seems to produce mostly K-feldspar. The Fe content from the clay minerals is not used for augite formation as in the yellow bricks, but is oxidized to hematite.

An interesting observation was made when looking at the reaction processes for the different clay types. It was found that the temperature at calcite and kaolinite break down (release of CO_2 and OH) is depending on the particle size. Smaller grains generally reacted at lower temperatures. This was one of the hypotheses to be tested in the project.

Main results of the dielectric characterisation and small-scale microwave heating trials (WP2), MPE

The main results are:

- The dielectric property measurements of clays establish that both clay types are within limits of reasonable microwave process ability across the firing range at the frequencies of 2450MHz and 911MHz. Moreover, both clay types have a similar dielectric response despite mineralogical differences.
- The dielectric property study of the major clay constituents reveals that most show a large degree of transparency to microwaves. Nevertheless, clay minerals with their moderate microwave absorbability dominate the dielectric response, where particularly muscovite and montmorillonite enable the potential for microwave processing.
- The Böttcher mixing rule was chosen as the base for the dielectric properties predictive tool for its accuracy, having an average estimation error similar to the measurement uncertainty of 5.1% for the dielectric constant and 18.8% for the loss factor. Thus, the dielectric properties of any clay mixture can be accurately estimated at any density and for the frequencies of 2450MHz and 911MHz from the dielectric properties of crushed mineral powders and the clay composition.
- Microwave firing (with no other heat source) of clay specimens at laboratory scale can produce fired products 92% faster than in conventional heating. Additionally, these sample size bricks have on average 11.5% and 13.43% higher compressive strength for red and yellow types than those conventionally processed.

WP2.1 Measurement of the dielectric properties of red and yellow Danish clays and their major constituents at different conditions of temperature, frequency and density

The results of the dielectric properties of carved chunks of red and yellow clays of the same density as raw bricks are presented in Fig. 11. It is clear that both clay types have a similar behaviour across the firing range, where the dielectric constants are largely unaffected by the temperature increase or clay composition. On the other hand, the loss factor is characteristically more sensitive, showing clear fluctuations that correlate with thermal transformations.

Free water moisture is the dominant dielectric phase below 100°C due to its stronger coupling with the microwave field compared with other clay constituents. Its effect is seen as a peak around 50°C in the losses. In the 100-350°C range, clays reach their highest degree of microwave transparency as their loss factors decrease as a result of the removal of physically bound water. Above 350°C, the start of clay minerals dihydroxylations and the associated continuous release of interlayer cations outside the mineral lattices cause a steady increase of the overall specimen's loss factor. An inflection point to the loss trends takes place around 800°C as sintering begins, due to the porosity reduction and the enhanced diffusion of charge carriers associated with this process. At this stage the thermal decomposition of calcite occurs. Interestingly, the elimination of CO_2 and resultant porosity increase competes with the sintering process causing a momentary cessation in the increase of the loss around 800°C for yellow clays. As a result of their calcite absence, red specimens become the lossiest from this point forward. Ultimately, all clays are well within the established boundaries of the generally accepted limits of microwave process ability, where the lowest loss factor of 0.026 more than doubles the accepted lower threshold of 0.010. Measurements at 910MHz give similar results. However, working at lower frequencies increases the influence of the ionic conductivity on the dielectric properties, resulting in an average 50% increase in the loss factors of red and yellow clays. On the other hand, decreasing the sample's density decrease their potential for microwave heating. The dielectric constants remain nearly the same in both cases.

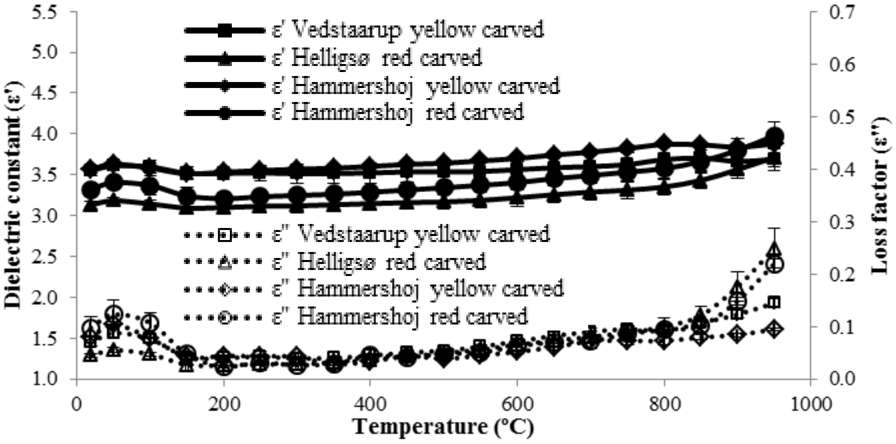

Fig. 11. Average dielectric constants and loss factors across firing between 20-950°C at 2470MHz for yellow and red Danish clays carved at 0.37±0.02 (Vedstaarup yellow), 0.39±0.01 (Hammershoj yellow), 0.40±0.01 (Helligsoe red) and 0.42±0.03 (Hammershoj red) void fraction.

The observed importance of the clay minerals phase on the dielectric response of the overall clay can be explained by the study of the dielectric properties of the single minerals seen in Fig. 12. Here, it is clear the non-clay minerals are essentially microwave transparent, meaning that the clay mineral phase, which becomes the dominant dielectric phase after free water elimination, is the one that enables the microwave treatment of clays. Particularly montmorillonite and to a lesser extent muscovite are the most microwave absorbent species, which occurs on account of their water absorption capabilities and presence of interlayer cations.

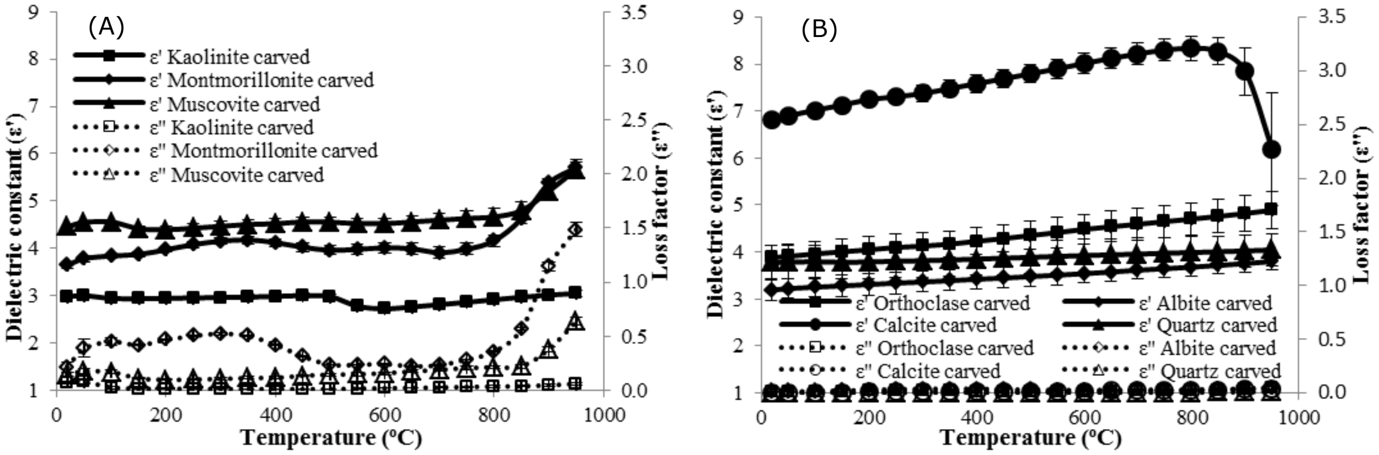

Fig. 12. Average dielectric constants and loss factors across firing between 20-950°C at 2470MHz for carved kaolinite at 0.48±0.02, montmorillonite at 0.32±0.03, muscovite 0.22±0.07 (A), orthoclase at 0.17±0.07, albite at 0.35±0.06, calcite at 0.09±0.03 and quartz at 0.17±0.01 (B) void fraction.

WP2.2 Development of a predictive tool capable of estimating the dielectric properties of a clay mixture from its composition using electromagnetic mixing rules

Using the dielectric properties of the same constituents of Fig. 12 as crushed powders as to avoid sampling issues, the estimation of the dielectric properties of red and yellow clays can be attempted. An example of the results is displayed in Fig. 13. All mixing rules provide trends akin to the measured values. Nevertheless, most of the approximations display large estimation errors, particularly in the loss factor prediction, except for Böttcher. This rule shows a degree of uncertainty of 5.1% for the dielectric constant and 18.8% for the loss factor, which are close to the resonant cavity measurements of Fig. 11. After validation at other frequencies and densities and with other European clays that display different dielectric trends on account to their lack of montmorillonite, this equation still provides accurate estimations of the dielectric properties of clays.

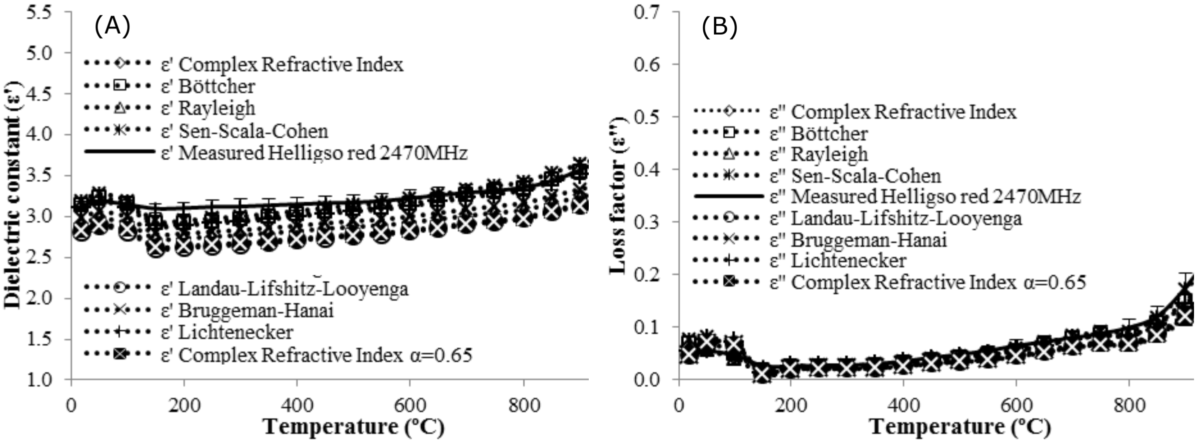

Fig. 13. Average dielectric constants (A) and loss factors (B) of measured carved clays and mixing rule approximations from powdered clay constituents across firing at 2470MHz for Helligsoe red clay.

WP2.3 Performance of laboratory scale microwave heating trials on red and yellow clays on heating cycles akin to those used in brickworks and assessment of product quality compared with conventional products

The results of WP2.1 were used to design a bench scale microwave firing apparatus, which is shown in Fig. 14. 25x20mm extruded clay cylinders were used in these experiments.

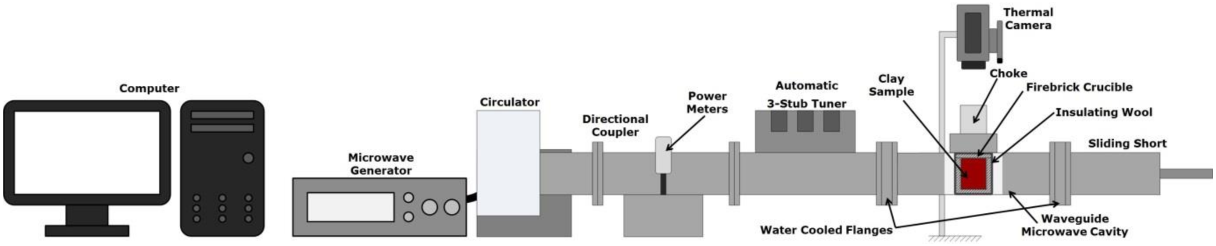

Fig. 14. Schematic of single mode microwave rig designed for microwave heating trials at 2450MHz. Sample and insulation are seen through cut in waveguide

The temperature profiles of the bench scale microwave heating trials on red and yellow clays are presented in Fig. 15. No cooling control was attempted due to the small sample size. Using a starting input of 200W, the top temperature is achieved in around 2h, less in the case of yellow clay. This is a result of exothermic reactions such as that of quicklime occurring after calcite decomposition, and causes sample melting unless tight power control is exerted. Due to the small size of the samples and power control limitations, slight overheating around 1050°C for yellow clay was unavoidable. Holding times of more than 30min invariably caused melting of the specimens.

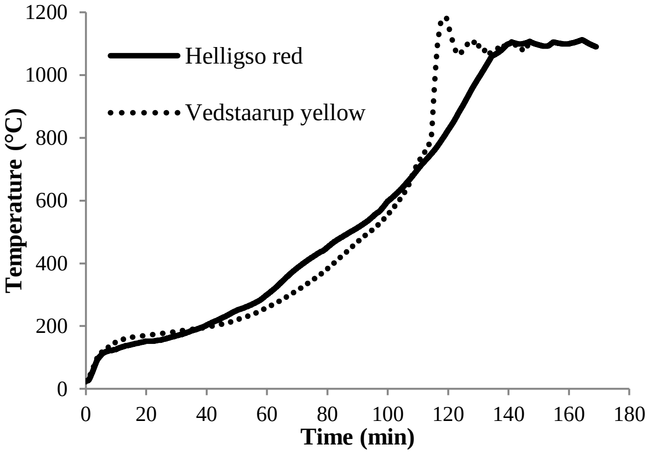

Fig. 15. Average clay temperature profiles from microwave heating trials on 25x20mm red and yellow clay cylinders at variable incident power controlled by their absorbed powers.

In order to enable the quality comparison of conventionally and microwaved clays, the same 25x20mm clay cylinders were conventionally fired. A common brickworks temperature profile was employed due to the sharp decrease of the brick properties at increasing heating rates. Table 1 shows a comparison of the performance of the microwaved clays of Fig. 15 and conventionally fired bricks, and Fig. 16 shows their appearance. Not only were the microwaved samples produced 93% faster than conventionally fired ones, but they also had on average a 12% higher compressive strength. Nevertheless, the shorter duration of the holding stage causes a dimmer coloration of the clay surface. Additionally, the microwaved products are more porous, as proved by their up to 50% higher water absorption, even though their saturation coefficient is comparable to the conventionally fired samples.

Table 1. Compressive strength and cold and hot water moisture absorption of microwaved and conventionally fired clay specimens presented as per ASTM C62.

	Vedstaarup yellow		Helligsoe red	
	Conventional	**Microwave**	**Conventional**	**Microwave**
Compressive strength (N/mm^2)	6.11±0.21	6.99±0.11	6.37±0.29	7.15±0.18
Maximum absorption cold water 24h (%)	9.5±0.3	15.4±0.3	7.6±0.2	10.7±0.3
Maximum absorption boiling water 5h (%)	10.5±0.2	16.5±0.3	8.9±0.1	12.1±0.2
Maximum saturation coefficient	0.90±0.02	0.93±0.02	0.85±0.01	0.88±0.02

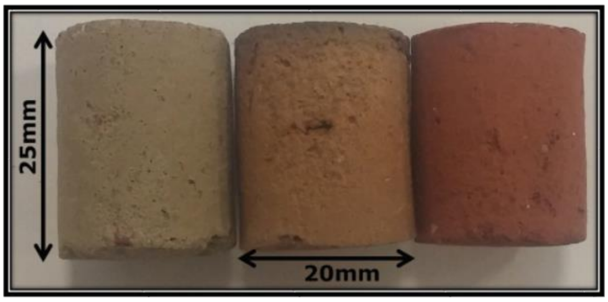

Fig. 16. Raw (left), microwave (middle) and conventionally (right) fired clay cylinders of 25x20mm

The conclusion of this work package is that small samples can be fired using 100% microwave energy, which surpasses the expectations set out by the original project plan. Thermal runaway occurred after the onset of sintering in these small microwave experiments. However, better temperature control may be possible with larger sample loads, and therefore it may be possible in the future to fire a full sized brick using 100% microwave energy. Nevertheless, in order to determine whether full sized bricks could be fired using microwaves alone, new design concepts for microwave firing cavities will be required.

It should also be noted that very fast heating may cause failure in the phase changes of minerals. A sign of this is seen in Fig. 16, where the microwave fired sample does not reach the red colour typical for the sample on the right.

WP2.4 Electromagnetic simulation

To speed up testing and to better understand the performance of the MAGF furnace, electromagnetic simulation work was performed in conjunction with the experimental work carried out by DTI (which is reported in WP3 and WP4).

The simulation work was underpinned by building a model of the MAGF-kiln and measuring the dielectric properties of the bricks at different temperatures from 20°C up to 1000°C to mimic the dielectric properties during firing. Simulation were carried out using COMSOL Multiphysics®, a finite element analysis simulation software. It allows the calculation of physical parameters such as electric field and power density, which are vital to establish the uniformity and efficiency of the microwave treatment. Simulations were performed at 2.45 GHz and input power of 1 kW per feed. The 3D model was meshed using tetrahedral elements.

The simulation work aimed to establish the electric field distribution and power density inside the kiln for the different load configuration. Fig. 17 presents an example load configuration reproduced from the kiln

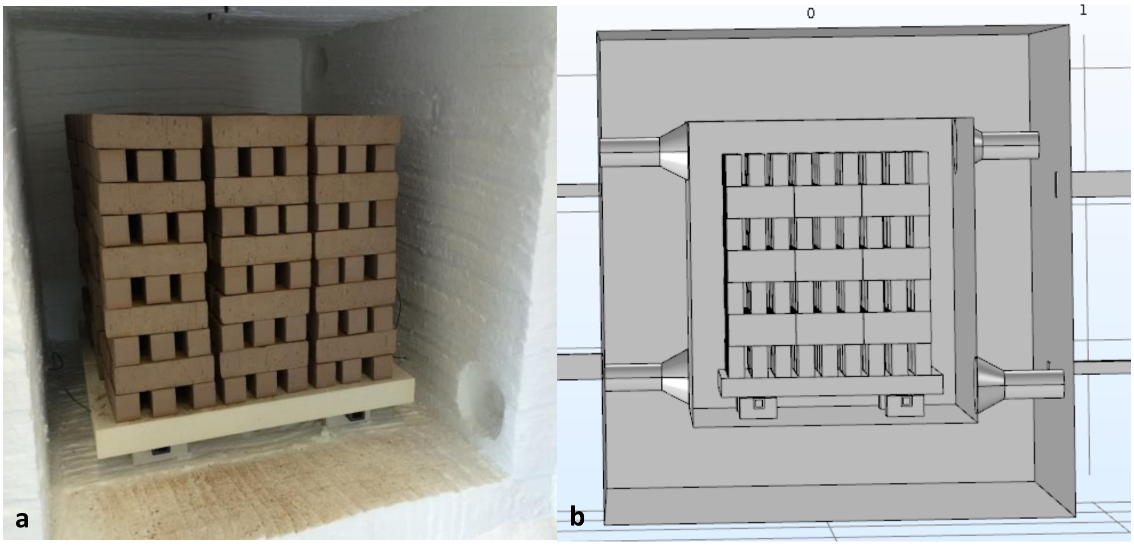

Fig. 17. Example of load configuration: a) MAGF kiln and b) reproduced in COMSOL.

Fig. 18 represents the electric field distribution inside the kiln with four feeds generating microwave energy to couple with the bricks.

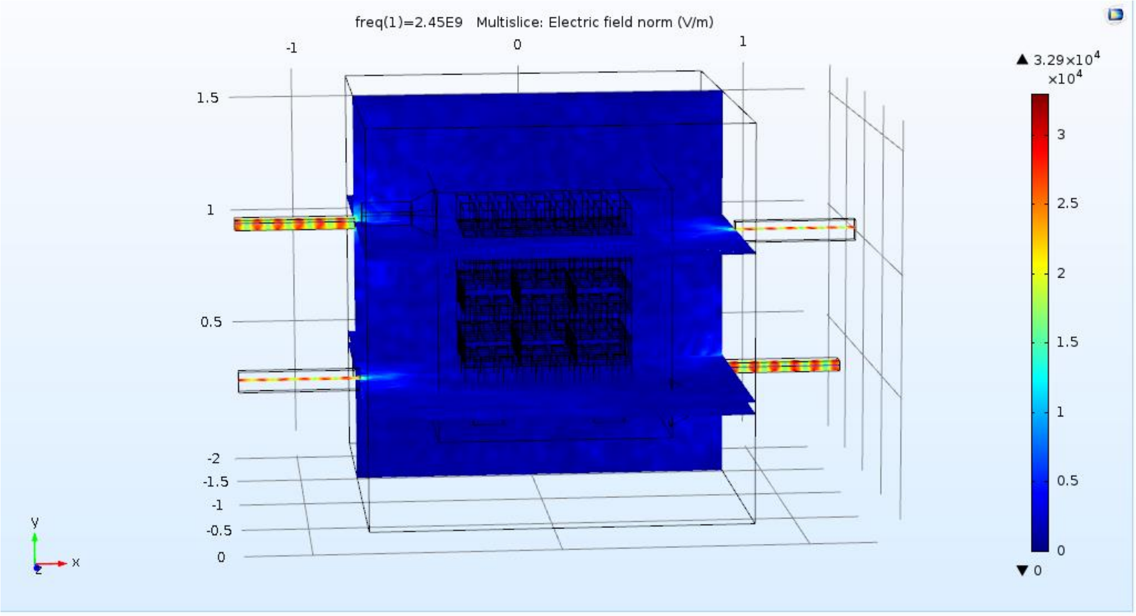

Fig. 18. Predicted electric field distribution inside the MAGF at 20°C.

Overall the electric field is evenly distributed throughout the load. However, a high field distribution (hot spot) is shown on the edges of the bricks. As the load is significantly large compared to the chamber this is challenging to adjust without the need to change the feed size and or the chamber design.

The electric field distribution allows calculation of the efficiency of the system (Table 2), which is described as proportion of the incident energy that is predicted to be absorbed by the load without the use of automatic tuning. During firing, automatic tuners are installed on the system as these devices allow the amount of energy absorbed into the load to be maximised. In the case shown in

Fig. 17, the system efficiency is better than 25% at room temperature and more than 70% at high temperature 900°C.

Table 2. Efficiency of the MAGF versus temperature

Temperature (°C)	System efficiency (%)
20	26
50	47
100	35
900	51

The outcomes of this simulation will help to establish the optimum configuration for treatment and allow further optimisation of the system for both red and yellow clays. As noted in Section 1.4, these simulations need to be validated with "real" dielectric property data, which is work in progress.

Main results of pilot plant and product development (WP3 and WP4)

The primary results can be summarized as follows:

- Established a pilot kiln for brick firing has been established with the MAGF technology at DTI.
- Established firing cycle for red brick "cold to cold", with the same dense stacking as used by the brickworks in the conventional tunnel kilns, with 39% microwave energy, and resulting in good product quality
- The above-mentioned firing cycle allows for a 40-45 % reduced heating and dwell time, compared with the conventional firing cycle (Cooling time cannot be reduced), potentially leading to a 33% increase in kiln throughput.
- Development of evaluation criteria and comparison with conventionally fired bricks
- Based on the optimal MAGF firing cycle, a 20-28 % energy saving and 50-55% reduction in gas consumption is shown relative to two baselines established in the pilot kiln, with gas firing alone
- An individual report including a roadmap for each brickwork has been prepared for a potential implementation of MAGF technology, which shows the potentials of the individual work.

The pilot plant consists of the MAGF kiln with its control system and associated water cooling system for microwave generators, chimney, gas tank systems and relevant safety features such as gas discharge monitoring. The kiln capacity corresponds to firing of approx. 200 pcs brick in Danish normal format. The control system enables the control of the temperature cycle "cold to cold" with gas alone, or with microwaves and gas (MAGF) in any combination where microwaves can represent up to 50% of the energy. The control is based on a temperature sensor at the top of the kiln. To achieve the desired temperature (set point), the 4 gas burners turn on or off automatically. The microwaves are programmed in optional steps from 0 to 2x6 kW, which is the maximum capacity. The temperature can be logged 4 places in the kiln with moveable thermocouples, and all energy consumption is monitored. Maximum temperature is approx. 1300°C, but this has not been tested, as the current materials in the project can withstand a maximum of 1100°C. The kiln is thus used to reproduce the same firing cycles as used in Danish tunnel kiln and develop optimized firing cycles with MAGF technology.

An important result of the experimental firings was, that it is possible to use the microwave energy fully during heating. However, microwave power needs to be lowered and more closely monitored during the dwell time, i.e. the time the peak temperature is maintained. The microwaves can assist with up to 50% of the energy and, according to the results, is limited only by the capacity of the microwave generators.

The fired bricks were assessed on the basis of standardized methods and partly on the basis of criteria established during the project to quickly determine whether a firing was successful. These are visible cracks, sound, hardness, and reduction cores: A tendency for darkening of the bricks' interior (see Fig. 6b), which indicates that the mineralogical conversion processes have not had enough time to elapse. It was suspected that the dark cores were caused by low oxygen contents during firing, but this is contested by the measurement of the oxygen content in the kiln atmosphere and of a test firing with a loss of oxygen.

With the use of microwaves, it is possible to increase the heating rate from 40°C per hour to 80°C. At the same time, the dwell time at peak temperature is reduced from 6 hours to 4 hours. The total time saving on a firing cycle is then 13-15 hours. Such a time saving will mean a significant increase in productivity on a tunnel kiln. If a cycle cold to cold lasts approx. 2 days, then a time saving on heating and dwell, cautiously estimated to 12 hours, will increase the efficiency of the kiln at 33%. Cooling time cannot be forced.

Energy savings of up to 28% per brick were obtained using MAGF technology in the pilot kiln, compared with gas firing according to conventional “slow” firing cycle. This is estimated by comparison of energy consumption of gas and MAGF firing cycles with similar number of bricks. The energy savings achieved cannot be directly converted to tunnel kiln production, as it is primarily attributable to the energy loss from the furnace, which is reduced by a shorter process time. Energy loss in a tunnel kiln per brick produced is somewhat smaller. The energy savings achieved by reducing heating time will depend on the energy efficiency and isolation of the oven in question. This is part of the assessment of the potential of each project partner.

The 3 roadmaps that are supplemented with data for the work's own clay etc. is delivered confidentially to the individual work. They contain in brief: mineralogy for clay samples and for fired brick, data for measuring dielectric properties. A summary in Danish of the project results. Description of the production plant and production range and amount. A suggestion for where MAGF is best placed. An assessment of potential benefits of using MAGF, including increased plant productivity and potential energy savings and CO_2 savings through the introduction of green energy.

Evaluation of test results, samples of fired bricks

To assess the quality of brick fired in the pilot furnace, samples were taken from selected firings, and compared with sample of bricks fired in tunnel kiln. A number of standardized properties were tested. An overview of the samples taken is shown in Table 3.

Table 3. Overview of test samples

Firing ID	Principle	Total no of bricks	Heating rate °C/hour	Dwell time hours	No of bricks in sample
Tunnel kiln	Gas only	n/a	40	6	18
Pilot-016	Gas only	72	40	6	10
Pilot -019	MAGF	72	100	2	10
Pilot -026	Gas only	72	100	2	10
Pilot -030	MAGF	180	80	4	18
Pilot -032	MAGF	180	80	4	18

The sample Pilot-016 is a "baseline" gas firing, with the same heating rate and holding time as tunnel kiln. In principle, we should achieve the same brick properties as when burning in the tunnel kiln. Differences between sample pilot-016 and sample from tunnel kiln can thus be attributed to the pilot kiln itself.

Table 4 shows achieved mean values for the samples. Standardized brick testing methods have been used. MAGF firings are marked green in the table.

The mean values generally do not show large fluctuations, except for water absorption, which is clearly lower for the sample fired in tunnel kiln.

Table 4. Mean values of selected properties, test samples listed in Table 3

Mean values brick samples	Length [mm]	Normalized compression strength [MPa]	Net density $[kg/m^3]$	Initial rate of water absorption $[g/m^2]$	Water absorption [weight%]	Pore filling rate
Tunnel kiln	228	20.7	1940	2.3	7.7	0.58
Pilot-016	230	24.3	1906	1.9	9.2	0.64
Pilot -019	231	23.7	1885	1.9	10.0	0.64
Pilot -026	232	25.0	1868	2.0	10.7	0.68
Pilot -030	233	24.7	1854	2.1	11.2	0.68
Pilot -032	233	26.2	1842	2.0	11.7	0.70
Total mean	231	24.1	1883	2.0	10.1	0.65

The mean values from the pilot firing vary considerably less than they differ from the mean values of brick samples from the tunnel kiln.

The difference between pilot-016 and the means of all samples from pilot firings is indexed in Table 5, where mean values from the tunnel kiln are set to 100.

Properties that show a difference greater than 10% are marked with light yellow. The highest value is highlighted with **bold.**

Table 5. Indexed mean values from Table 4

Indexed mean values	Length	N. compression strength	Net density	Initial r of w abs	Water absorption	Pore filling rate
Tunnel kiln	100	100	100	**100**	100	100
Pilot-016	101	117	98	83	119	110
Mean of all pilot firings	101	**119**	96	84	**137**	**115**

Pilot-016, which has the same firing cycle as the tunnel kiln, is very close to the mean of all pilot firings regarding length, compressive strength, net density and initial rate of water absorption. Only the results for water absorption and pore filling rate show for pilot-016 a larger deviation from the other pilot firings, being nearer to the results from the tunnel kiln bricks. This shows, that the firing cycle cannot be copied exactly from the tunnel kiln to the pilot kiln, resulting in exactly the same properties of bricks. It is therefore reasonable to make the assessments of the various firings in the pilot kiln, solely by comparing with pilot-016, since pilot-016 is assumed to be the baseline corresponding in principle to conventional gas firing.

Table 6 shows, where relevant, preferred values for the selected properties.

Table 6

Characteristic	Generally Preferred values for Danish bricks
Length [mm]	Within declared value ± 3 mm
Normalized Compressive strength [MPa]	Above declared value. For loadbearing masonry 20 MPa would be sufficient in most cases
Net density $[kg/m^3]$	No specific limits
Initial rate of water absorption (MS) $[g/m^2]$	Preferred values are $1,0 < MS < 3,0$ $[g/m^2]$
Water absorption [w%]	No specific limits but lower values are preferred
Pore filling p_f	$P_f \leq 0,8$ is considered frost resistant (facades, Norway) $0,8 < p_f \leq 0,9$ is considered "uncertain" $0,9 < p_f$ is considered not frost resistant

Overall, the bricks from the pilot kiln have a greater length and higher compressive strength, while the net density is smaller and water absorption and the pore filling rate is higher. Apart from the higher compressive strength, these numbers correspond to the fact that the brick from the pilot kiln generally have had a lesser shrinkage than the bricks from the tunnel kiln. This may indicate that certain mineral phase changes have elapsed differently in the kilns, even with similar firing cycle. The lower compression strength found in the tunnel kiln bricks is most likely the result of the cooling process, with compressive strength being the only property that can be affected during cooling. Conclusively, all bricks from the selected firings have achieved satisfactory mean values that do not vary dramatically.

However, to get an impression of the effect of faster heating rate and the use of MW, the pilot firings are compared to each other. In Table 7, the values for selected firings are indexed and compared.

Table 7. Indexed mean values from table 4

Indexed mean values	Length	N. compression strength	Net density	Initial r of w abs	Water absorption	Pore filling rate
Mean 19&26	101	100	98	103	**113**	103
EUDP-016	100	100	100	100	100	100
EUDP-019	100	95	101	95	93	94
EUDP-026	100	**100**	100	100	**100**	**100**
EUDP-030	100	94	101	105	96	97
EUDP-032	100	**100**	100	100	**100**	**100**

Comparing pilot-016 relative to a mean of pilot-019 and -026, which both have the same rapid heating and short dwell time, indexing indicates that the rapid heating has affected mainly the water absorption. As an average, water absorption has increased by 13% by fast heating and short dwell time, which is not desirable.

Then pilot-019 and -026 are indexed with pilot-026 = index 100. Since pilot-019 is fired with MAGF while pilot-026 is gas alone, the effect of MAGF seems to be a slightly lower compressive strength, water absorption and pore filling rate. The bricks fired with MAGF are a little weaker, but probably more frost resistant.

After completion of the rapid firings with small loads of 72 bricks, pilot firings were scaled up with larger loads of 180 bricks. Here it was necessary to reduce the heating rate to 80°C per hour and extend the dwell time to 4 hours in order to achieve uniform firing of the load.

By indexing pilot-030 (MAGF) and pilot-032 (gas), a similar effect can be noted as when comparing pilot-019 and -026. The trend is the same, but the differences are a little less for water absorption and pore filling, namely that the MAGF fired bricks have a lower compression strength, and at the same time lower water absorption and pore filling rate, meaning better frost resistance.

Based on the test results, it is assumed that the proportion of microwave energy can be increased, if the firing rate is lowered. This is however not readily possible, since the temperature control is based on the gas burners, not the microwave. It was not foreseen when designing the pilot kiln, that more than 50% mw energy would be possible

Results and expectations on turnover, exports and employment

DTI has employed 1 extra person as a direct result of this project, and expects increased turnover as well as some export, based on training courses/consulting on MAGF and conversion, product development, Product testing and pilot runs – also with conventional gas firing.

For the brickworks there is no immediate results from the project in the terms of increased turnover, exports or employment, since the MAGF technology is not ready for direct implementation. The individual roadmaps show potential for increasing process efficiency, energy savings and the possibility to convert from fossil fuels to green energy sources, which would lead to an increase in turnover and exports and eventually increased employment as a result of an improved competitiveness.

These items are very relevant for each partner, and each partner has expressed keen interest in the further development of the MAGF technology.

For the university partners, AU and UoN, as well as C-Tech Innovation there are no immediate results in the form of increased turnover or employment. But the partners see a potential in future R&D projects, as described in section 1.7 Perspectives, which will lead to increased turnover and/or employment. This could very likely be in the form of export, since there is a market for the technology throughout Europe.

1.5.2 Dissemination

Dissemination of the projects result will take place in four major steps:

1. Information targeted at the Danish brickworks through meetings and reports that convey the most important results of the project and interpret the potential of Danish brickworks
2. Articles in relevant media
3. Participation in conferences and meetings
4. Through consulting on commercial terms and homepages.

Originally, an international conference at the end of the project was planned. The delay in the project explained in section 1.4 did not affect the basic performance of the project, but the ambition to hold an international conference had to be abandoned, as the results have been so late in the project that there has been no time to plan one.

Therefore, the plan to hold a conference was abandoned at the January 2017 steering group meeting, as this would take too much effort in this late stage of the project, where focus should be on achieving the main objects and on recovering the delay of the project. Instead the participation in conferences is planned.

Targeted information

In 2018, DTI will visit the Danish brickworks (12 in total) and/or invite them to meetings at DTI. There will be the main results present related to the potential of the MAGF technology of the individual brickwork/organisation.

During the project, information has been presented to the Danish Brickworks through the industry organization “Danske Tegl” and the professional network “Dansk Teglmesterforening” at several meetings:

- 2017-05-10 to 13, participation in study tour of Dansk Teglmesterforening in Holland
- 2017-10-13, open house at the DTI, presenting (among other things) the pilot kiln, a total of 70 visitors
- 2016-10-27, Danske Tegl, member’s reunion, presentation, project progress
- 2016-09-24, Dansk Teglmesterforening, yearly reunion, presentation of the project
- 2015-11-05, Danske Tegl, member’s reunion, presentation, project progress
- 2015-11-19, Dansk Teglmesterforening, yearly reunion, presentation of the project.

Articles and conferences

- Sustainable manufacturing of next generation building materials using microwave energy, Miguel Calvo-Carrascal, Eleanor Binner, Juliano Katrib, Sam Kingman, *Ampere-15th International Conference on Microwave and High Frequency Heating, 2015*
- Sustainable manufacturing of building materials using microwave energy, Miguel Calvo-Carrascal, Eleanor Binner, Juliano Katrib, Sam Kingman, *Frontiers in Materials Processing Applications, Research and Technology*, *2017*
- Towards Sustainable Manufacturing of Building Materials: Prediction of Dielectric Properties and Physical Transformations in Complex Clay Mixtures, Miguel Calvo-Carrascal, Eleanor Binner, Juliano Katrib, Sam Kingman, Ole Bjørslev, Lars Peter Salmonsen (authors tbc), to be submitted to *Ceramics International*
- “Microwave assisted gas firing”, in Ziegelindustrie International, no. 08, Abelone Køster
- “Nu skal mursten brændes med mikrobølger”, article in Ingeniøren 2014-09-01 (description of project start, objectives etc., by journalist Ulrik Andersen).

Consulting

DTI will distribute the new knowledge and technology at the Danish brickworks as well as in Sweden and Germany on commercial terms through consulting. The pilot plant will be the Institute's launch pad into the emerging markets and will complement the commercial business in the process consultancy to the brickworks in Denmark.

https://www.teknologisk.dk/ydelser/murvaerk-og-tegltage/keramisk-produktion-med-mikroboelger/22658,8?cms.query=magf

https://www.teknologisk.dk/ydelser/nu-skal-tegl-braendes-af-mikroboelger/35307?cms.query=magf

In cooperation with UoN:

https://www.dti.dk/industrial-microwave-processing/industrial-microwave-processing/35701

1.6 Utilization of project results

The results of the project can be utilized in several contexts where volumetric heating i.e. microwave heating provides a quality and / or energy advantage. Hence the project results will, once the MAGF technology is matured and implemented, contribute to realize energy policy objectives.

Phasing out fossil fuels

The Danish Government's energy strategy 2012-20 states, that the total Danish energy supply (electricity, heating, industry and transport) is to be converted to renewable energy by 2050. Danish brickworks are among the most energy-intensive industries; in 2002 the total energy consumption at the plants amounted to 2,08 PJ (Energisparekatalog for teglværksbranchen, 2003). This corresponds to approx. 1,6% of all industrial energy consumption.

The DK government has a goal to phase out the use of all fossil fuels by 2050, and there is an increasingly critical view of the current use of fossil fuel in the industry. The conversion to renewable energy sources is therefore essential but is not possible without an introduction of new technology.

Conversion to electricity may be accomplished by the use of dielectric heating where the energy from the microwaves is brought directly into the goods.
A shift to renewable energy at the brickworks is therefore an important milestone of the energy agreement ambition to liberate the dependence on fossil fuels. The results of this project has shown the potential for shifting at least 50% of the energy consumption for firing to microwave energy, and has created the knowledge base for further maturing and implementing the technology in brickworks.

In addition to the potential of MAGF technology, the project has also produced ground-breaking new knowledge of raw materials and phase changes during brick firing.

Hence, exploitation of project results will be done in several areas

- Implementation of MAGF technology in Danish brickworks
- New knowledge for expanding the consulting business for DTI and C-Tech
- Platform for new R & D projects, further development
- Education at universities
- Marketing of brick products.

Implementation of MAGF technology in Danish brickworks

The new knowledge will play a key role in the brickworks' expected conversion of energy source in the coming years. The results of the project will be utilized to facilitate a smooth transition into production. However, as described above, the technology needs further maturing /development, before this can happen.

The knowledge will be offered as courses and consulting, and commercial development projects to the target group of Danish and foreign brickworks. Directly following the end of the project, DTI will visit the brickworks to present the results and discuss the potential for each brickworks.

Based on the results of the scientific research, pilot production at DTI will facilitate the implementation in the industry. This forms the basis for an upscale to the production plants of the participation companies. C-tech Innovation, is a possible supplier of both consulting and of refurbishing existing kilns from natural gas firing to MAGF technology.

New knowledge for expanding the consulting business for DTI and C-Tech

DTI will commercialize the results by supplying consultancy, as well as pilot plant production to scale up at the brickworks participating in the project, with a later focus on the markets in Denmark, Sweden, Norway, Germany and the Netherlands.

C-Tech will provide design consultancy on the integration of microwaves into advanced kiln systems. This will build on the materials selection safety and process optimisation experience from the background and the development work undertaken in the project. This work will cover several territories including Europe and North America and India, where there has been investment in new technology and efficient manufacturing technologies.

Marketing

The industry in focus is highly challenged in the building products market because consumers are starting to look critically at the energy consumption for the production of bricks. A halving of production energy will enhance the image of the products enhancing competitiveness in international markets

The brickworks will commercialize the results by enhanced competitiveness because of lower energy costs and lower production time, as well as through new improved products and colours.

Education (AU Geoscience)

The firing of clay mixes to brick is a very illustrative example of mineralogical changes under different temperature conditions. This is comparable to geological processes such as contact metamorphism of clay-rich sediments. The projects approach, its development, and application has been used as an example in courses like mineralogy to show the application of knowledge acquired by the students during natural science educations such as in Geoscience. Moreover, the research component of the project is extended to future projects on brick production.

This project included a PhD studentship for Miguel Calvo at UoN. It has provided him with a high quality educational experience by enabling him to: work on a project with genuine industrial application; work in world class laboratory facilities with a multidisciplinary supervisory team; spend time in an overseas facility (DTI); attend regular meetings of international industrial and academic partners, and present his work at two international conferences. Miguel in fact received second prize for his poster at the *Frontiers in Materials Processing Applications* conference. The microwave heating equipment and waveguide measurement techniques that Miguel has designed and built as part of this project will go on to be useful for other students in related areas at UoN.

Product development, in general, pilot plant

Based on the projects results, the brickwork’s core product development will be cheaper and safer, as the scope of "trial and error" experiments decreases as the method goes from experience-based to knowledge-based development.

Brickworks will then achieve lower costs, greater supply certainty, freer innovation of colour and more robust bricks for bricklaying.

AU and DTI will be submitting an application for an R&D project directly following the end of this project, which in part is based on knowledge about phase changing diagrams from this project.

1.7 Project conclusion and perspective

1.7.1 Conclusions

Mineralogical investigations:

1. Variations in the clay size fraction has significant influence on the reactivity and availability of elements (H1 confirmed).
2. For each raw material and their chemical composition, it is possible to model mineralogical phase changes as a function of temperature applied in the firing of bricks (H2 confirmed).

MAGF experimental firings:

3. With MAGF it is possible to convert up to 44% of the energy for firing to electrical energy, thus potentially green energy. (H3 is partially confirmed). An even higher proportion may be possible, but it was not possible to demonstrate this with the pilot kiln.
4. With MAGF it is possible to increase the heating rate and with the more uniform heating shorten the duration of the firing time. (H3 is partially confirmed).
5. The heating rate and the dwell time are not only determined by the ability of the kiln to heat the stack uniformly, but also that mineralogical phase changes require a certain amount of time to elapse.

Dielectric research and modelling:

6. It is possible to predict the dielectric properties for a clay mixture, based on the knowledge of the individual elements, with dielectric properties predictive tool, developed in the project.
7. It is possible to model the dielectric field in a given kiln and stacking using COMSOL Multiphysics®.

Elaboration of conclusions

Re 1:

Based on the extensive studies of raw materials and production mixtures, it is found that while there is a large variation in the grain size distribution of raw materials, this is levelled off in the production mixes where several types of raw materials are mixed and sand added. This picture is seen when focusing on 3 fractions;

- Clay, grain size <2 μm
- Silt, grain size 2 μm to 63 μm
- Sand >64 μm

However, the new knowledge about the importance of grain size for reactivity applies to grain sizes <2 μm, distinguishing between the fractions fine <1,3 μm and coarse 1,3-2,0 μm, an area that has not previously been studied and which may be relevant to the development of new clay mixtures adapted to MAGF burning. As noted under 5), certain phase changes are so slow that the potentially very fast heating with MAGF causes the clay to not undergo the necessary phase changes, as seen in the small samples fired with MW alone. Therefore, knowledge of the importance of grain size for phase development is important in future clay mix development and adaptation to MAGF firing. Its effect on drying should also be considered.

Re 2:

With the creation of new phase change diagrams, a crucial new understanding of phase changes has been achieved during the firing of clay to bricks. (see section 1.5). The only existing studies on the field (The typical Danish brick, a phase analytical study by x-rays, Ejnar Jensen, 1978) conclude for both red and yellow bricks that 30-35% of the fired brick consists of amorphous material or so-called "melt" or glass in red burning clay. Our study has not identified these volumes of amorphous material and the new phase diagrams provide a significantly better understanding of the transformation of clay minerals.

Re 3:

This is a very significant conclusion and a great progress, as previous pilot projects only operated with 10% MW of energy.

Re 4:

Experimental firings have shown that the heating rate can increase significantly and the dwell time can be shortened from 6 to 4 hours, as its function in practice is to ensure that the entire stack in the kiln reaches the same top temperature. It halves process time for the heating and dwell time in the firing cycle, which typically means a 13-15 timer shorter process time (compared to a typical tunnel kiln, 33% more throughput).

Re 5:

However, it is also a significant discovery that uniform temperature alone it is not sufficient to obtain good results, it must also be ensured that there is time enough for mineralogical phase changes to take place.

Re 6:

With the ability to predict the dielectric properties of a mixture, the need for experimental firings is significantly reduced, as these can be estimated from an analysis of the output minerals and phase diagrams.

Re 7:

Electromagnetic simulation and modelling of the di-electric field of a kiln, which was an additional activity, was not completed within the framework. But showed in principle the possibility to design kilns, stacking, refurbishing etc.

1.7.2 Perspectives and potentials

Phasing out fossil fuels (3), (7)

The conclusions (3) and (7) underpin the potential of MAGF technology to convert from fossil fuels to renewable energy sources. The project did not identify a limit for the proportion of microwave energy regarding product quality. The proportion of microwave energy was instead limited by the pilot kiln. Laboratory experiments indicated that firing is possible with 100% microwave energy in principle, but some practical problem with cooling of the surface must be foreseen in industrial scale. Conversion to electricity may be accomplished by the use of dielectric heating where the energy from the microwaves is brought directly into the goods. A shift to renewable energy at the brickworks is therefore an important milestone of the energy agreement ambition to liberate the dependence on fossil fuels. Although the technology level at the end of the current project has reached 6, the potential has clearly been shown (3) and the tool for modelling the dielectric field (7) should in future projects be utilized for refurbishing existing kiln or optimizing the design of new kilns, including the optimum stacking pattern for bricks and/other ceramic goods.

Energy savings (4), (5), (1)

It has not been possible to give a precise prediction of energy savings using the MAGF technology. There are two reasons for this. Firstly, within the limits of the current project, it was not possible to fully optimize the MAGF or gas firings in the pilot kiln, since the focus was on product quality and obtaining as high a percentage of MW energy in the firings as possible. Secondly, it is not possible to compare directly the energy consumption of the pilot kiln with the highly optimized (energy wise) tunnel kilns at the brickworks.

But there is no doubt, that based on the shortened firing and holding time alone (4), a substantial energy saving can be obtained in reducing the loss of heat from the kiln during firing. However, it must be considered, that there is a limit to how fast the firing can be (5). This could possibly be counteracted by utilizing the knowledge about the effect of grainsize (1) provided the raw materials can be found and/or manipulated.

TBE (Tiles and Bricks of Europe) calculates the total production of bricks and roof tiles to 56.9 million tonnes in 2010. This corresponds roughly to an annual energy consumption of 150 PJ. In addition, there are other ceramic industries.

In comparison, the Danish production is only a few percent of this, but Denmark is a technological pioneer in energy efficient production, and with this project a knowledge is achieved that can be utilized in the international market for energy efficiency and conversion to green energy.

Higher productivity (4), (5), (1)

The shortened firing cycle lead to a higher throughput by 33% potentially and hence, better productivity for the given kiln. The firing and/or the drying is typically a bottleneck in the manufacture of bricks. Therefore, the potential speed up of firing cycles and even drying (not investigated in this project) is very interesting for the brickworks, and is the most likely driver for the MAGF technology to be further developed and implemented in the industry.

Estimates from the pilot project in the UK calculates the payback period of investment in a new MAGF plant at 12 to 15 months with an investment of approx. 18 million DKK. Calculations are based on energy prices of 0,19 DKK per kWh, and to halve the length of the tunnel kiln. Alternatively, it is possible to convert existing plants to MAGF.

Optimizing product quality (1), (2), (5)

The projects main focus was on ensuring that products fired with as high a share of microwave energy as possible, had at least the same quality as bricks fired with conventional technology. Therefore, the potential for optimizing product quality has not yet been explored. The new insight into mineral formation at different temperatures (2) and in terms of grain sizes (1) allows for predicting where MW has the best effect in the firing process. It has not come as a big surprise, that mineral phase conversions actually can take a long time. With the conventional gas firing, where the achievement of uniform temperature throughout the stack takes some time there will be enough time for phase changes. Documentation of the time needed for phase change (5) will be an important parameter in optimizing product quality.

Developing new products and/or new clays mixes (2), (6)

Development of new products through the knowledge of mineral formation at different temperatures allows for predicting where MW has the best effect in the firing cycle.

Future possibilities include clay mixes with special additives for enhancing microwave firing and utilization of waste products: An interesting perspective is the ability to add agents with special dielectric properties in the desired temperature ranges (6).

There is enormous potential for "green products" as well as for development and consultancy services in the ceramics industry. Currently, production of building bricks in Denmark uses only half the capacity of approx. 450 million bricks. New technology will make it possible to increase competitiveness by halving the processing time, enabling a doubling of production at lower costs. While the Danish production is quite small compared to the European market, it is realistic to expect a sharp increase in exports, with competitive prices due to the results of this project and the attractive special yellow brick, which is unique to Denmark. If the entire potential output growth in Denmark is exported, this represents almost a 10-fold increase of current exports, or approx. 1,2 billion DKK for the entire industry.

Optimizing MAGF kilns and refurbishing existing kilns (7)

The ability to model the microwave field in an oven is a powerful tool in the design of new furnaces, and the assessment of the possibility of combining an existing furnace with microwave generators. This includes the optimal way to stack the bricks in the kiln. It should be possible to expand the simulation tool with flow simulation, so that the movement of the air in a MAGF oven can be included in calculating the optimum temperature distribution.

Once the dielectric data have been validated for real bricks, the model will be updated, and therefore the results obtained from the simulation work package will be able to be cross-compared with the experimental work performed by DTI to establish the working conditions of the MAGF and propose a forward plan to treat bricks at maximum efficiency and better product quality.