Processing with Microwaves at High Temperature for the Industrial Sector

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The use of microwaves in the industrial sector

Microwave heating is used routinely in the domestic and catering sectors for cooking and reheating a variety of foodstuffs. However, what is the penetration of microwave heating in the industrial sector? There are routine applications such as tempering of foodstuffs, drying of pharmaceuticals and drugs under vacuum and rubber curing. Other notable applications include cooking of bacon, drying of pasta, drying of complex automotive parts or sludge and in plasma etching or enhanced CVD operations. Its sister technology of RF heating, has routine installations for drying of textiles, moisture levelling of biscuits and in plastics welding.

The above applications take place at relatively low temperatures as contrasted to applications of microwaves at high temperatures such as the processing of ceramic and other materials where surprisingly the penetration into the industrial sector has been extremely small. The latter sector can be considered as an emerging technology, where its potential has not yet been fully exploited despite a concerted effort for the past thirty years.

In all potential microwave applications, it is necessary to consider the nature of the material, which involves the understanding of any variation or transformation taking place by the constituents as well as the processed material itself during exposure to the intense electromagnetic fields, within the applicator system.

For such applications to be successful, one has to pay special attention to applicator design, the monitoring and control systems, dielectric properties of the material as well as put in place protection and safety means thus resulting in an efficient industrial application.

A systemic approach is needed for the processing of material with microwave energy. In the specific case of high temperature furnaces[1], the following aspects should be considered:

- The system conditions for heating the material.
- Material transformation due to the heating.
- Changes in the overall conditions of the system during the transformation
- Capacity to modify the conditions during the transformation.
- Monitoring and identification of changes during the treatment.

The dynamic interaction of the materials processed with high electromagnetic fields alters the spatial temperature distribution, which may subsequently lead to transformations within the materials as well as variations to their properties. The induced changes during microwave heating of mineral materials at high temperatures[1], may include mass transfer, decomposition, chemical reactions, recrystallization, textural changes, as well as other physical chemistry phenomena.

Some of the changes during microwave heating are progressive, such as the molecular vibration due to the high temperature (which also leads to variations in the microwave absorption) while other changes may take place abruptly. Consequently, data of the dielectric properties during the process are needed as one of the fundamental inputs for a proper design of both the applicator and the process.

Our contribution to industrial applications

The proposed systemic approach to industrial microwave applications, may lead to new opportunities in the market when processing at high temperatures. In particular, CEINNMAT is focused in material processing in continuous flow using microwave and hybrid systems for industry (or yet for the scalability from the laboratory). Some applications are also developed directly for materials characterization.

The development of a microwave application combines the knowledge of the specific application with the relevant material science, as follows:

- Study of the material transformations during the application of microwave power, and evaluate the chemical and physical transformations and properties modifications.
- Active and passive materials in the configuration of the kilns.
- Solution of material transport during microwave heating.

The portfolio of CEINNMAT consists of a large number of potential applications. In this paper we highlight the treatment at high temperatures in raw material processing (Fig. 1). Each application developed is dependent on the material to be treated, as well as to the specification of each process. It makes the microwave technology a typical tailor-oriented development. Eventually, the raw material is also treated to facilitate the microwave treatment[3].

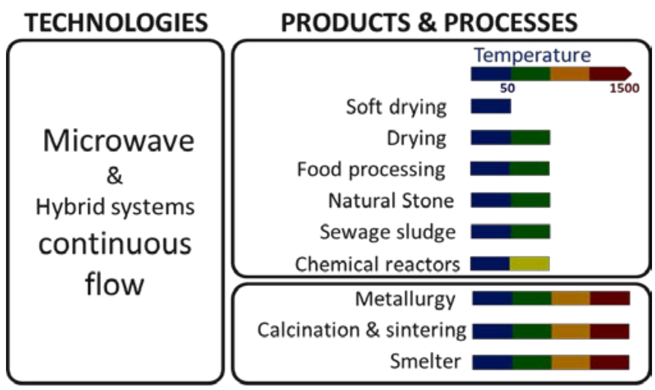

Figure 1. Proprietary technologies developed by CEINNMAT and partners, in different levels of implementation (at TRL 5-9).

To guarantee the successful application, a previous study to adapt the process to the solution must be performed, considering the treatment requested, as well as the industrial parameters, such as, production speed, quality and cost-efficiency. It is evident therefore that potential microwave applications in industry require a multidisciplinary approach.

We have developed several new technologies offered to the market (Fig. 1), including high temperature for calcination, sintering and melting, but also other specific applications with the knowledge of the process, as follows:

- Chemical reactors: a continuous microwave reactor with good homogenous heating and effective control of power.
- Complex applications in continuous processes: Restrictive and demanding applications, such as, homogenous temperature profile and control, very soft drying, ultra-fast drying, curing of resins and multi-materials treatment. Figures 2 and 3 show examples of planar materials which are non-confidential in the pilot stage[3].
- Metallurgy: A unique technology for extractive and advanced metallurgy (Fig. 4) where factors other than heating are also produced and exploited.

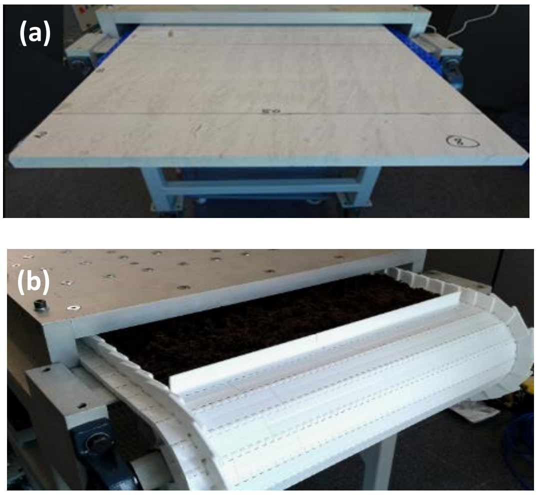

Figure 2. Examples of microwave applications (a) for flat materials, and (b) for granular materials with a conveyor belt for a cost-effective low temperature continuous flow.

Prior to the implementation process but also as part of its development, CEINNMAT offers customers the service to examine the full feasibility and implementation study so as to minimize the risk in the new technology.

CEINNMAT is working in cooperation with customers and a wide group of complementary research institutions concerned with microwave, materials science, engineering, electronic and control, robotic as well as processing to assure the quality of the study, as our collaboration with the ITACA Institute.

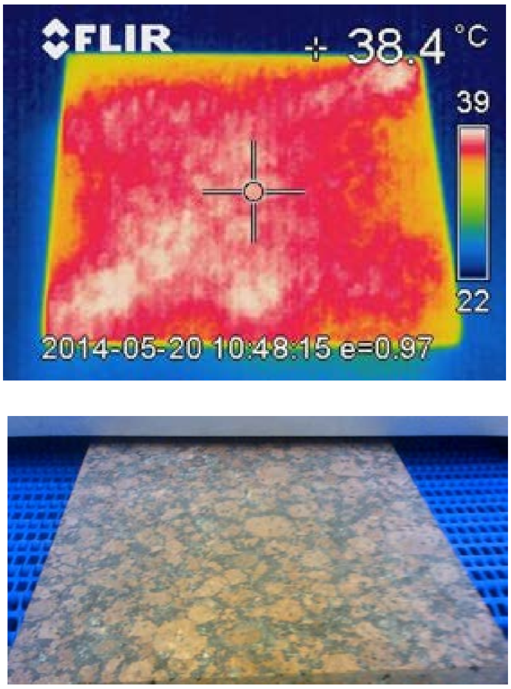

Figure 3. Example of application of microwave in granite slab industry.

Figure 4. Example of application in metallurgy processing (proprietary technology).

Calcination of silicates with microwaves

The industrial applications of calcined and fired silicates are very wide, particularly in the ceramic and cement industries, but also in the pharmaceutical and in the food industries.

Calcined clays are widely used in cement industry, where they are produced to replace parts of the clinker content as supplementary cementitious materials (SCM) and hydraulic lime because they exhibit pozzolan activity.

Fired clays are used in the ceramic industry, usually between 890 ˚C and 1250 ºC but also up to 1700 ºC, to obtain sintered ceramic materials.

The evaluation of the material response to the microwaves is needed to design and adjust the microwave kiln in order to obtain a successful industrial production. Apart from the knowledge of the mineralogical reactions, the dielectric properties characterization with temperature is essential for the industrialization of the microwave-assisted calcination and firing processes.

Figure 5 shows a dielectric thermal analysis heated using microwave energy (MW-DETA) of a kaolinitic illite clay sample[1]. A powdered sample was analysed in a dual mode cavity, using two independent microwave sources for heating and measurement, developed by DiMaS at ITACA Institute[5]. A cylindrical sample holder filled with 1.2 cm^3 of clay was heated and monitored for complex permittivity and temperature measurement by optical pyrometer. The clay material was heated up to primary mullite formation (Mu) at 1000 °C for characterization. After minor restructuration by losing crystalline water of clays, the major mineral transformation starts with the structural disordering by dehydroxylation. At higher temperatures, the crystal reorganization is competing with the amorphous glass formation.

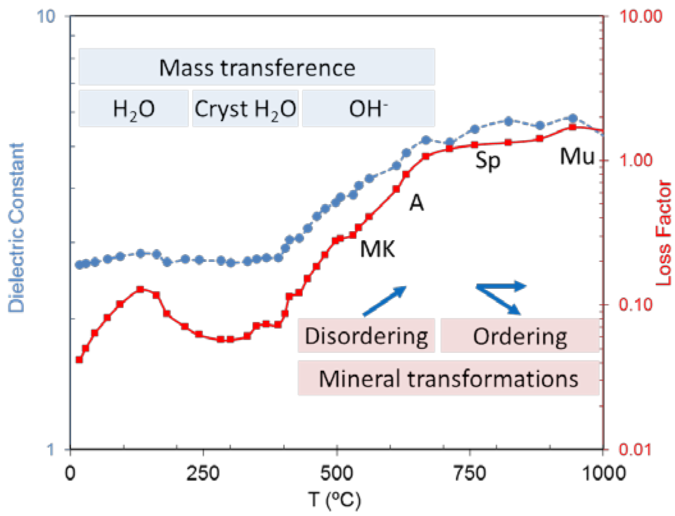

Figure 5. Microwave dielectric thermal analysis (MW-DETA). Dielectric constant (blue) and loss factor (red) vs. temperature during heating in a kaolinitic illite clay.

During the heating of clays, one can identify a sequence of changes in the domain of mass transfer changes and mineral transformations, as follows:

(1) Free-water evaporation evidenced by the loss factor increasing during its phase change;
(2) Crystalline water sublimation;
(3) Dehydroxylation of kaolinite and liberation of OH- groups, hence microwave absorption.

In the example shown in Fig. 5, a transformation from kaolinite mineral to an amorphous metakaolinite (MK) is evidenced. The disordering continues with the temperature, with the presence and changes in the amorphous phases (A). Minor reorganization during transitional spinel (Sp) formation show small decreasing in the loss factor and dielectric constant. The formation of Mullite as major crystalline ordering is evidenced from 950 °C measured up to 1100 °C.

Figure 5. Rotary kiln interior during silicate calcination

A new pilot plant will be constructed for different applications to have it available for trials, as well as the service for full scale trials for existing or new applications. Cooperation of the end-user with our research team is strongly recommended in order to develop and implement successful applications. In some applications, we would also welcome additional qualified partners.

For further reading:

1. López-Buendía AM, García-Baños B, Bastida J, Llorens-Vallés G, Urquiola MM, Catalá-Civera JM. (2016) "Microwave calcination of clays". 3rd Global Congress on Microwave Energy Applications (3GCMEA). Cartagena (Spain), July 2016.
2. López-Buendía, AM; Garcia-Baños, B; Urquiola, MM; Gutiérrez, D; Catalá-Civera, JM (2016) "Dielectric non-destructive testing for rock characterization". Natural Stone Industry and Cultural Heritage. EGU2016 European Geosciences Union 2016.
3. López-Buendía, AM, Urquiola, MM, Arizo, A., García-Baños, MB, Plaza, P, Catalá-Civera, JM (2015) "Dielectric measurement for rock characterization and microwave heating prediction". 15th International Conference on Microwave and High Frequency Heating. Proceeding paper AMPERE 2015.
4. Garcia-Baños, B, López-Buendía, AM, Suesta, C, Catalá-Civera, JM, Jiménez-Reinosa, Fernández, JF (2015) "Dynamic study of microwave heating of quartz sand up to 1100°C and effects of particle size". 15th International Conference on Microwave and High Frequency Heating. Proceeding paper AMPERE 2015.
5. Catala-Civera, JM, Canós-Marín, B, Plaza- González, P, Gutiérrez Cano, JD, García Baños, B, Peñaranda-Foix, FL, (2015) "Dynamic measurement of dielectric properties of materials at high temperature during microwave heating in a dual-mode cylindrical cavity," *IEEE Trans. Microw. Theory Tech.*, **63**, 2905-2914

About the Author

Angel López Buendía, PhD, CEO in INNCEINNMAT SL since 2012, BSc, MSc in Geosciences (Unv. Granada, 1989), PhD in Geology (Univ. Zaragoza, 1995), research technician (Univ. Valencia) post-doc Researcher in Univ. Leicester, UK), with background in petrology, mineralogy and geochemistry. He has worked in mineral exploration and material sciences. During 2001-2012, he was the director of teams and large installations in research institutions in the field of material science, material processing, geomaterials and geometallurgy. He has been member on standardisation in

CEN and RILEM committees. Dr. Buendía has been the expert representing the Spanish delegation to the NMP program of FP7 in 2010. Expert of the AIAG of the PPP-EeB for FP7 and the Roadmap H2020, and member of the Operational Groups of the EIP of Raw Materials in Horizon 2020. He has been a member of the scientific societies of Geology (EGU, SGE, AEQUA) and microwave technology (AMPERE). He has co-authored 30 peer-reviewed scientific papers and 14 patents. CEINNMAT has won some awards and recognition for the microwave systems developed as the innovative SME seal (Spain), Excellence Award seals (European Commission), TOP3 Sustainability Award (Germany) for the project DAPhNE, FP7 314636, in which CEINNMAT has the role of industrial microwave kiln developer.