# Development of Closed Porous Microcellular Products from Perlite

A. Peppas, M. Taxiarchou and E. Koffa,
*National Technical University of Athens, Greece*

T. Karalis and A. Amanatidis
*S&B Industrial Minerals S.A., Greece*

ABSTRACT

Perlite is a naturally occurring siliceous rock, which can be expanded from four to twenty times its original volume when heated at a temperature close to its softening point. The expanded perlite is characterized by increased sound and thermal insulating properties. However, the conventionally expanded perlite products have high porosity and low mechanical properties that limit the range of their applications in the construction and chemical industry. The present work aims at the development of new closed porous microcellular material from perlite with enhanced mechanical and physical properties to be applied in new applications, like joint compounds, functional fillers for special paint types, special blasters and mortars, artificial marble. For the production of these new materials a vertical electrically heated furnace with six different heating zones and controlled gradient temperature profile was used. The effect of major process parameters, like temperature and air flow, on the quality of the produced materials was studied using Greek perlite from Milos Island. The quality of the new products was evaluated in terms of their bulk density, compression strength, water and oil absorption, surface area and pore volume. The experimental results show that the produced materials are characterised by improved mechanical and physical properties compared to the conventionally expanded perlite products.

## 1. INTRODUCTION

Perlite is a volcanic origin industrial mineral with high silica content; it also contains low amounts of alumina, sodium, potassium, very minor amounts of impurities and a water content varying between 2 and 5%. In Greece perlite deposits have been found until now on the Islands of Milos, Kimolos, Kos, Gyali and Antiparos. The largest quarrying activity in Greece and European scale occurs on the island of Milos in Aegean Sea.

When perlite is rapidly heated to a range of 800-1400$^{o}$C is explosively expanded as the contained water flashes to steam, and form a light weight commercial product. The escaping steam generated by the rapid and relative non-uniform heating of the perlite particles in the heat source and blasts through their plastic walls, resulting in the production of expanded particles with numerous cracks and fissures. These characteristics give to the final product high water and oil absorption and low compression strength.

The conventional expansion of perlite ore is usually accomplished by feeding ground, dried, pre-sized perlite ore into a vertical chamber that contains a direct flame at its bottom end directing a forced air flow upwardly. The ore particles are typically introduced to the hottest region of the expansion chamber at or near the flame at a temperature of 1450$^{o}$C (Papanastasiou, 1979).

Temperatures vary significantly across the chamber, with the highest temperatures been observed in the area in and near the flame. The average residence time of perlite particles in the expansion chamber is relatively short (milliseconds) and in most cases is generally less than one second, except for the coarsest particles (Sodeyama, et al. 1999).

The conventionally expanded particles are chemically inert, resistant to relatively high temperatures and are characterised by considerable sound and thermal insulating properties. Due to these properties, they have a wide variety of industrial applications such as horticultural aggregate, high weight insulating construction products and numerous other uses where a light weight, granular filler material is useful. However, their applications are somehow limited due to the high degree of open porosity and the lack of durability or stability. Durability or stability refers to their characteristic that although the expanded particles may begin as hollow particles when used in liquid systems or liquid environments, eventually lose much of their hollowness due to the penetration of the host liquid through pores and fissures of the expanded particles. This phenomenon becomes more intense due to the breakage of the rather soft expanded perlite particles. The host liquid penetrates and essentially fills the originally hollow expanded particles, which in turn lose their lightweight property because their cavities are no longer filled with air, but are filled or nearly filled with host liquid. When such filling takes place, the density and the viscosity of the liquid and perlite particles mixture significantly increase (William, 1988).

For these reasons expanded perlite is inferior compared to non porous synthetic particles, such as glass, ceramic, or plastic hollow microspheres for applications that require durable and lightweight fillers. Several technologies have been developed to overcome this problem of liquid penetration aiming to the coating of the expanded particles with silicon coatings like dimethyl silicone, dimethydichlo-rosilicane, titanates and zirconates.

## 2. EXPERIMENTAL SETUP

### *2.1 Overview*

In order to be able to produce lightweight expanded perlite with substantially closed porous spherical particles a pilot scale vertical furnace (Fig. 1) was set up at the Laboratory of Metallurgy of the National Technical University of Athens aiming at the controlled heating and residence of perlite particles in the reactor chamber. The furnace uses electrical resistances as heating source and includes a retort, a volumetric screw feeder, an air hammer and a particle collector.

The raw perlite is discharged directly at the top of the furnace using a volumetric screw feeder at a rate of 0.1 to 1 kg/h. A stream of air flow at a rate of 5-200 l/min is injected at the direction of gravity force, using a remote conventional air compressor, in order to control the particle residence time and the furnace temperature. A collection chamber has been installed at the bottom in order to collect the hot expanded particles.

The expansion chamber is made of an Inconel pipe of 13.2 cm diameter and 3 m length, suitable for electric furnace applications. The choice of the alloy was based on its ability to withstand the high temperatures of the furnace environment, as in the case of conventional expansion furnaces, and its corrosion resistance. A small air gap has been established between the outer wall of the expansion chamber and the inner surface of the heat source in order to prevent displacements in the electrical heat elements and to ensure heat transfer by radiation from the heating elements to the expansion chamber walls.

The heat source is made of KANTHAL alloy and has a total heating capacity of 24 kW. It is divided in six zones, each one comprising two electrical resistances, and it is insulated within a layer of Al-Si-O refractory material. Conventional thermocouples have been placed within the furnace, between the expansion chamber and the heating elements, for temperature control and monitoring of each heating zone. Near the bottom of the furnace and positioned against the expansion chamber,

an air hammer has been installed in order, through vibration, to loose particles which stick to the inside wall of the expansion chamber when it operates at high temperatures. Excessive particle build up on the walls results in the creation of poor thermal and flow characteristics in the expansion chamber and may even lead to flow blockage in extreme cases.

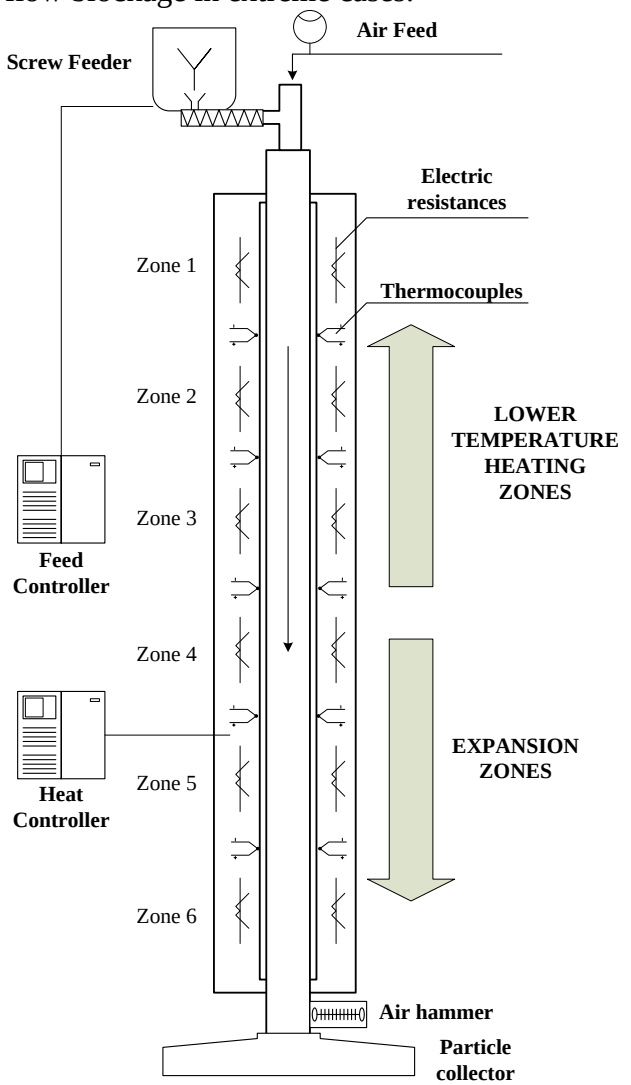



Figure 1: Expansion furnace setup

The expansion procedure in the furnace is based on the indirect heating of the particles through radiation of the expansion chamber walls. Heating can be controlled so that all perlite particles can be exposed at uniform and relatively lower temperatures and for higher residence time in the expansion zone than in the conventional processes. This controlled process permits the particle walls to become soft and remain plastic during the expansion, so that the expansion does not occur explosively leaving a porous surface, but occurs quite slowly and uniformly to form a rather closed porous surface over the entire periphery of the expanded particle.

### *2.2 Materials used and characterisation methodology*

A perlite ore from Milos Island, Greece (S&B Industrial Minerals S.A.) was used for the expansion experiments. The specific gravity of the material was 2.25 g/cm$^3$, the loss of ignition 2.82%, the specific area 0.31 m$^2$/g and the total pore volume 0.54x10$^{-3}$ cm$^3$/g with an average pore diameter of 7.05 nm. The chemical analysis of perlite in main elements is presented in Table 1.

Table 1: Chemical analysis of perlite used on expansion experiments

| Oxide | % w/w |
|---|---|
| $SiO_2$ | 76.38 |
| $Al_2O_3$ | 10.64 |
| $TiO_2$ | 0.14 |
| $Fe_2O_3$ | 1.05 |
| MgO | 0.19 |
| CaO | 1.29 |
| $Na_2O$ | 4.27 |
| $K_2O$ | 3.22 |
| L.O.I | 2.82 |
| Total | 100 |

X-ray diffraction (XRD) analysis showed that the studied perlite sample consists mainly of amorphous silica, but also contains quartz, feldspars and illite.

The particle size distribution of the studied perlite (Fig. 2) was determined using a Malvern Laser Particle Analyser for particles with a size of -500 μm and sieves for the bigger fractions. The analysis showed that the sample has a mean size ($d_{50}$) of 190.23 μm.

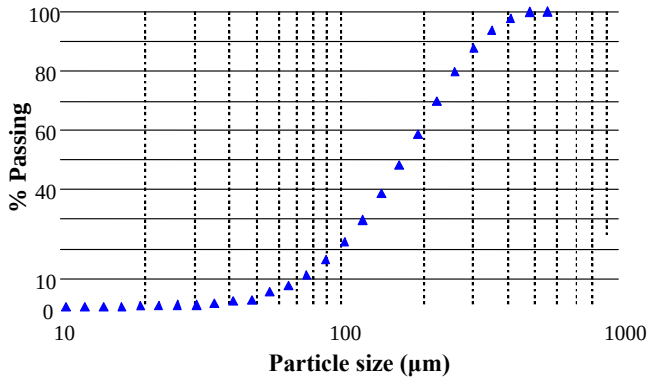



Figure 2: Particle size distribution of raw material

Moreover, differential thermal analysis (DTA) was carried out using a SETARAM-TG/DTA 92-16 apparatus by heating the sample in helium atmosphere in the range of 20-1100$^{o}$C with rate 5$^{o}$C/min. Two endothermal peaks were determined in the range of 200-400$^{o}$C and at 570$^{o}$C, which are attributed to the removal of crystalline water the and transformation of traces of a-quartz to b-quartz (Fig 3).

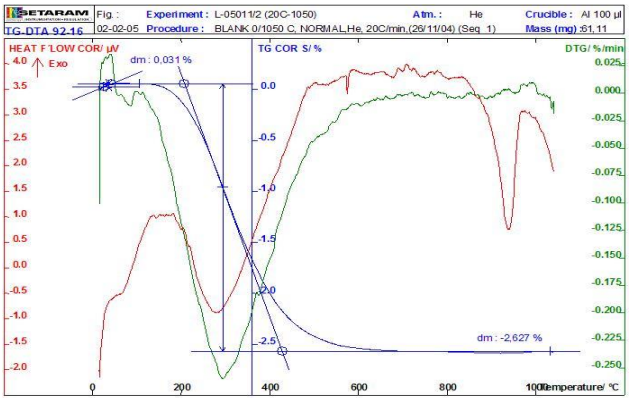



Figure 3: TG and DTA analysis

For the characterisation of the expanded products L.O.I, oil and water absorption, compression strength and sinks/shattered/ floaters distribution were determined. The morphology and the geometry of the particles were also studied through Scanning Electron Microscopy, using a JEM 1600 analyser. These are the main properties specified by the end users of relative commercial products.

The measurement of oil absorption was based on the ASTM standard D 1483, "Standard method for oil absorption of pigments by Gardner-Coleman Method" (ASTM, 2002). The water absorption method is a customised procedure developed by perlite producers, based on DIN EN 1097-6, aiming at the determination of the water volume absorbed in the perlite sample after a saturation and drying cycle (S&B Industrial Minerals S.A, 2001). The compression strength measurement is based on the calculation, through a hydraulic ram, of the pressure established at the surface of the sample when the sample height has been reduced by one inch (S&B Industrial Minerals S.A, 2003). The sinks (non expanded particles), shattered (semi expanded particles) and floaters (expanded particles) distribution was determined through the volume calculation of each fraction in a Imhoff cone, where a mixture of 600 ml of water and 300 ml of the expanded sample has been added and gently stirred. The floaters percentage, as specified from the end users, should be more 90%.

### *2.3 Samples produced*

In total three types of samples were produced. In order to be able to compare the properties of the materials expanded by the conventional and the controlled process, an expanded sample was prepared in a conventional lab scale vertical furnace at S&B Industrial Minerals S.A. premises. Moreover, four isothermal experiments were performed at 750, 850, 950 and 1050$^{o}$C using the controlled process in order to estimate the temperature required for the expansion of the studied perlite at a bulk density of 90 to 120 kg/m$^{3}$, which is a target range in the electric furnace. Finally, in order to evaluate the effectiveness of the controlled expansion procedure three samples were produced in the electric furnace at different temperature profiles. The feed rate of the perlite ore in all cases was 1 kg/h and the air flow varied from 20 to 30 l/min. Based on the geometrical characteristics of the expansion chamber it was calculated that this air flow corresponds to a speed of 2.44 and 3.65 cm/sec respectively. The complete experimental conditions are presented in Table 2.

Table 2: Experimental conditions

| Sample | Air flow (lt/min) | Zone temperature (°C) 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|---|
| Conv.-122 | | Flame temperature (1450°C) | | | | | |
| Isoth.-750 | 20 | 750 | 750 | 750 | 750 | 750 | 750 |
| Isoth.-850 | 20 | 850 | 850 | 850 | 850 | 850 | 850 |
| Isoth.-950 | 20 | 950 | 950 | 950 | 950 | 950 | 950 |
| Isoth-1050 | 20 | 1050 | 1050 | 1050 | 1050 | 1050 | 1050 |
| Control-1 | 20 | 800 | 850 | 900 | 975 | 1050 | 1100 |
| Control-2 | 20 | 850 | 900 | 950 | 1000 | 1050 | 1150 |
| Control-3 | 30 | 750 | 800 | 850 | 960 | 1130 | 1200 |

## 3. RESULTS AND DISCUSSION

The properties of the materials produced by the conventional and the controlled expansion process are summarised in Table 3 and 4.

Table 3: Main properties of the expanded products

| Sample | Bulk dens. ($kg/m^3$) | L.O.I. (%) | Oil absorp. (gr/gr) | Water absorp. (gr/gr) | Comp. strength (psi) |
|---|---|---|---|---|---|
| Conv.-122 | 122 | 1.69 | 3.34 | 3.61 | 42 |
| Isoth.-750 | 833 | 1.67 | - | - | - |
| Isoth.-850 | 482 | 1.48 | - | - | - |
| Isoth.-950 | 179 | 0.88 | 2.11 | 1.4 | - |
| Isoth.-1050 | 92 | 0.73 | 5.31 | 4.52 | 42.9 |
| Control-1 | 124 | 0.58 | 3.15 | 3.34 | 66.95 |
| Control-2 | 123 | 0.34 | 2.90 | 3.11 | 68 |
| Control-3 | 87 | 0.24 | 5.31 | 4.52 | 42.9 |

Table 4: Sink, shattered, floaters distribution of the expanded products

| Sample | Sinks (%) | Shattered (%) | Floaters (%) |
|---|---|---|---|
| Conv.-122 | 2 | 4.67 | 93.33 |
| Isoth.-950 | 1.5 | 3.83 | 94.67 |
| Isoth.-1050 | 0.5 | 5.83 | 93.33 |
| Control-1 | 0.83 | 8.5 | 90.67 |
| Control-2 | 1.5 | 3.83 | 94.67 |
| Control-3 | 0.39 | 3.16 | 96.45 |

The produced controlled expanded materials (Control-1, 2, 3) present significantly improved compression strength in comparison to the conventionally expanded product (Conv.-122) (the maximum improvement was 62%). The controlled expanded materials are also characterized by marginally decreased water and oil absorption, when compared to the conventionally expanded product at certain bulk density (122-124 $kg/m^3$). The loss of ignition of the conventionally expanded product was quite higher than this of the controlled expanded material, a property relative to the residence time of the particles in the expansion chamber. The floaters percentage was higher than 90% in all cases. The particle size analysis showed that the sample Control 1 has a mean size ($d_{50}$) of 136.36 μm (Fig 4), quite lower in comparison with the raw material indicating that the raw material particles were separated in smaller ones during expansion.

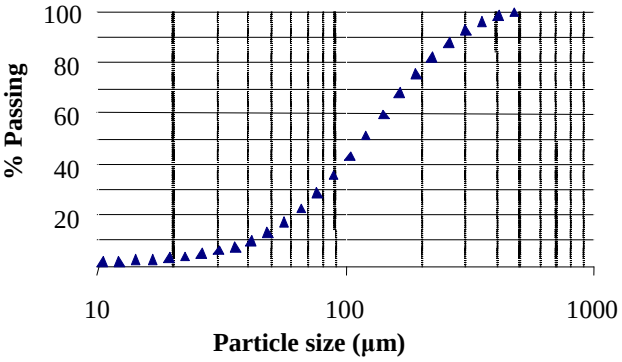


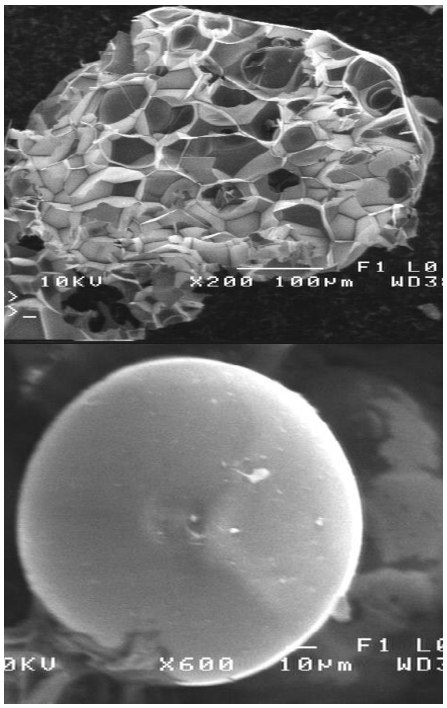
Figure 4: Particle size distribution of Control-1 sample.

The lower value of L.O.I derived from the controlled expansion at maximum temperature 1200°C in comparison with the isothermal expansion at 1050°C, where two expanded materials of the same bulk density were produced, showed the necessity of the upper lower temperature zones, where the particles walls become soft and plastic, i.e. of lower viscosity. A temperature value of 1050°C in the first zone of the isothermal test was quite adequate for the particles expansion.

The critical factor that affected the properties of the expanded products was the temperature profile. An increase by 50°C at all temperature zones (Control-2 vs Control-1 samples) resulted in slightly improved properties without affecting the bulk density. A reduction of 50-100°C at the upper temperature zones, combined with a

further increase by 50-100ºC at the lower zones (Control-3 vs Control-1 and 2 samples), worsened the properties, while improved the bulk density and the floaters percentage. Therefore, the maximum temperature of the heating zones is also critical, depending on the targeted property. The air flow rate was quite low to affect the expansion process; however a quite higher value may result in a significant temperature drop in the expansion chamber.

The production of hollow, closed porous products can be noticed on Scanning Electron Microscopy pictures, where the controlled expanded particles have a spherical shape and a relative smooth, continuous surface in contrast with the conventionally expanded perlite particles which have a quite different morphology (Fig 5).




**Conv.-122**
Conventionally expanded product

**Control-2**
Controlled expanded product

Figure 5: Geometry and morphology of the expanded materials

## 4. CONCLUSIONS

The continuous monitoring and control of residence time and temperature in the expansion chamber may result in the production of expanded perlite particles with more or less spherical shape, characterised by higher than usual compression strength (durability) and probably marginally lower open porosity. This procedure can be achieved using a vertical electrical heating furnace. Preliminary laboratory scale data, derived from the controlled expansion of Greek perlite samples, indicated that the compression strength was improved by 62% in comparison with conventionally expanded products. The data also indicate a slight improvement in terms of water and oil absorption, by 13%.

In order to study the main parameters, the performance and the optimum efficiency of the controlled expansion process, additional experiments are currently under preparation. The main aim of these tests is to develop an experimental model which, under typical operating parameters, will predict the efficiency of the expansion process.

## REFERENCES

ASTM Standards (2002), Standard method for oil absorption of pigments by Gardner-Coleman Method, D1483-95.

Papanastasiou D. J., (1979), Perlite expansion in a vertical furnace – a simplified theoretical analysis, Perlite Institute Annual Proceedings (Dubrovik): 67-71.

Sodeyama, K. Sakka, Y. et al (1999), Preparation of fine expanded perlite, Journal of Materials science, C9906 0215 476 Bd. 34 H10.

S&B Industrial Minerals S.A (2003). Method for durability measurement of expanded perlite one inch compression strength, Internal Test Procedure.

S&B Industrial Minerals S.A (2001). Method for water absorption measurement of expanded perlite, Internal Test Procedure.

William R. White (1991), Expanded mineral particles, apparatus and method of production, United States Patent, Pat.Num.5,002,696.

## ACKNOWLEDGEMENTS

The work has been financed by the General Secretariat for Research and Technology of Greece within the frame of International Cooperation in Industrial Research and the support is gratefully acknowledged.