# **An Introduction to Metakaolins**

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# **An Introduction to Metakaolins**



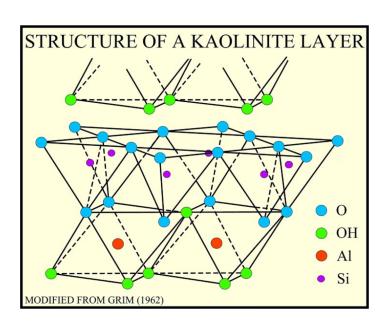
## What is Metakaolin?

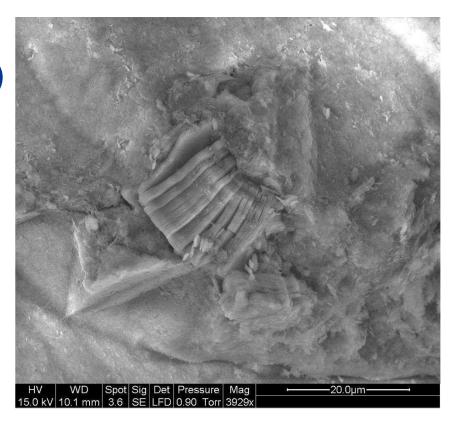
The Manufacture of Metakaolin
The Properties of Metakaolin



#### What is Metakaolin? -- The structure of kaolinite

■ Kaolinite is a hydrous aluminium silicate: Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub> or Al<sub>2</sub>O<sub>3</sub> . 2 (SiO<sub>2</sub>) . 2 (H<sub>2</sub>O)

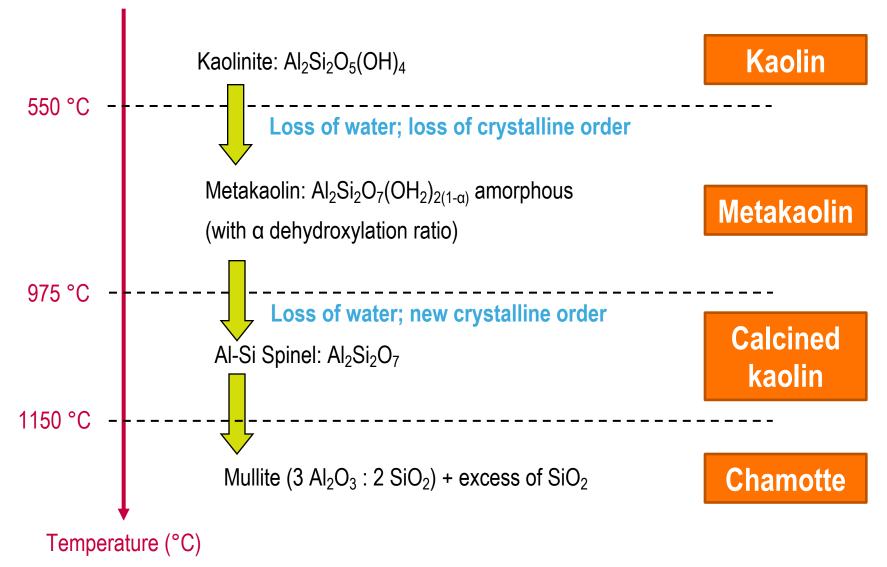




With heat, water is released from the crystalline structure and the structure is modified.

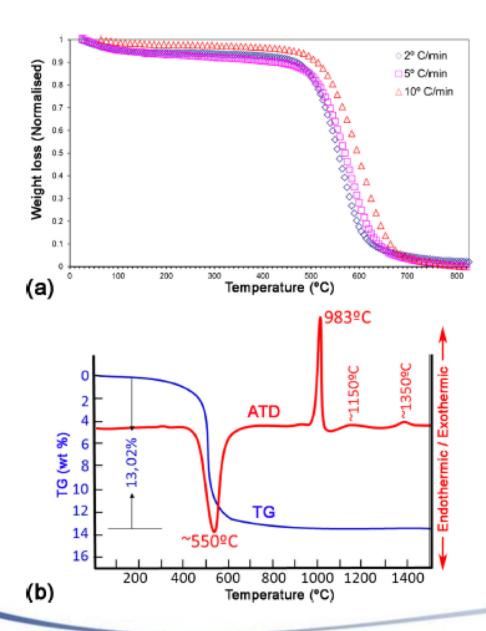


#### What is Metakaolin? -- The transformation of kaolinite





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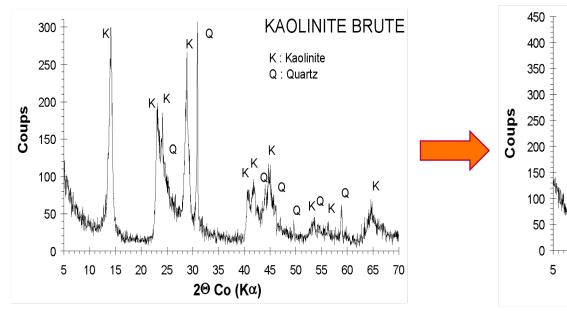
The DTA curve shows a broad endothermic peak at 550°C and a sharp exothermic peak at 983°C.

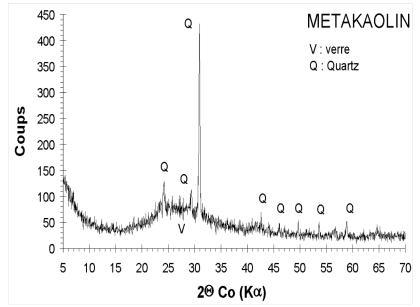
The main endothermic peak  $(\sim 550 \circ C)$  is associated with the loss of weight on the TG curve.



## What is Metakaolin? -- Structural characterisation

XRD shows the disappearance of the crystalline structure of kaolinite. The structure of metakaolin is amorphous.







## What is Metakaolin? -- Structural characterisation

<sup>27</sup>Al NMR shows the presence of Al<sub>V</sub>, a five-coordinate species. This species is very reactive and is characteristic of metakaolin.

|                      | Kaolinite   | Metakaolin   |
|----------------------|---|--|
| Structure            | <ul> <li>Al<sup>3+</sup> in octahedra.</li> <li>Si<sup>4+</sup> in tetrahedra.</li> </ul> | <ul> <li>Destruction of Al<sup>3+</sup> octahedra.</li> <li>Si<sup>4+</sup> in a polymer of tetrahedra.</li> </ul>       |
| <sup>27</sup> AI NMR | • Al <sub>VI</sub> signal (–3 ppm).   | <ul> <li>Al<sub>VI</sub> transforms into<br/>Al<sub>IV</sub> (70 ppm)</li> <li>&amp; Al<sub>V</sub> (35 ppm).</li> </ul> |
| <sup>29</sup> Si NMR | • Si <sub>IV</sub> signal (–90 ppm).  | • "metakaolin signal"<br>(–100 ppm).   |

#### Thermal transformations of kaolinises

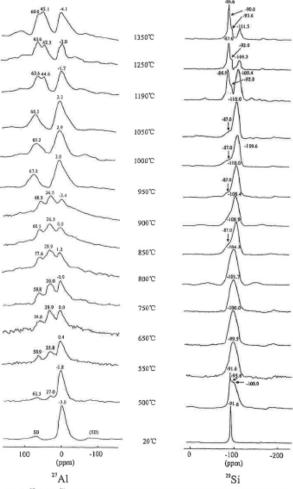


Fig. 3. 27Al and 29Si MAS NMR spectra of the knotinite heated at different temperatures.



# **An Introduction to Metakaolins**



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The Manufacture of Metakaolin

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## **Manufacture of Metakaolin**







#### **Feed Kaolin**

- Mining
- Preparation



#### **Calcination**

- Rotary Kiln
- Flash Kiln



## Milling

- Ball Mill
- Hammer Mill
- Roll-press Mill

#### Key performance factors

- Mineralogy
- Chemistry
- Specific Surface

#### Key performance factors

- Time
- Temperature
- Redox conditions

#### Key performance factor

Particle size

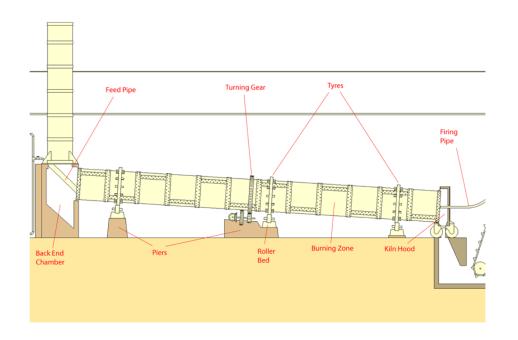


#### Manufacture of Metakaolin: Processes of Calcination

- There are several industrial processes to calcine a kaolin clay, some known since Antiquity.
- Two main processes used within Imerys:
  - <u>Continuous furnaces:</u> wherein loads are moved through temperature zones continuously of intermittently
    - Herreshoff multilevel kiln: UK, USA
    - Rotary kiln: France (Clérac), Ukraine (Vatutine), USA (Andersonville)
  - Flash kilns:
    - Torbed calciner: UK
    - Flash kiln FCB type: France (Clérac)



## Manufacture of Metakaolin: Rotary Kiln



www.cementkilns.co.uk

Residence time: 4 hours

Rotary kiln at Imerys Refractory Minerals Clérac (France)

L= 34 m; ø 2,5 m

Throughput: 10 tonnes per hour

Fuel oil + Biogas + Sawdust



## Manufacture of Metakaolin: Rotary Kiln

#### **Advantages**

- The technology is reliable and robust (similar to a cement plant).
- Efficient energy consumption: 800-1200 kWh/t
- Good throughput rate: 10-12 tonnes/h

#### **Drawbacks**

- Dehydroxylation control after heating: need to have a good knowledge of the process.
- The feed material is shaped as pellets
   → temperature gradient in the pellet.
- Product has to be milled after calcination.
- The kiln has to be run continuously so need of a certain volume or combination with other materials.

#### Product available:

- ARGICAL M-1000 (France)
- MK-40 (Ukraine)



#### Manufacture of Metakaolin: Herreshoff Kiln

#### **Advantages**

- Technology is reliable and robust (similar as rotary kiln).
- Efficient energy consumption: 600-1200 kWh/t
- Very good control of temperature of calcination

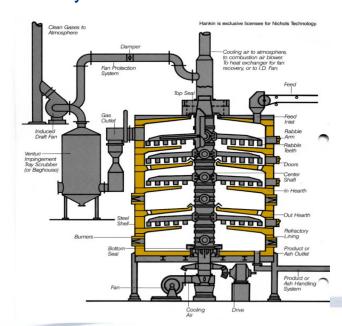
Residence time: 40 -- 60 minutes

## Product available:

❖ METASTAR 501 (USA)

#### **Drawbacks**

- Dehydroxylation control after heating: means to have a good knowledge of the process
- Thermal inertia of the kiln
- Huge investment: 1.5-2 times more than a rotary kiln.





#### Manufacture of Metakaolin: Flash Kiln

#### **Advantages**

- Really flexible: Target temperature quickly reached.
- Precise control of temperature, thus of dehydroxylation.
- Limited energy consumption: 400 to 800 kWh/t
- Capacity of kiln adapted by initial design (1 tonne/h at Clérac).
- Can produce very fine metakaolin (pre milling).

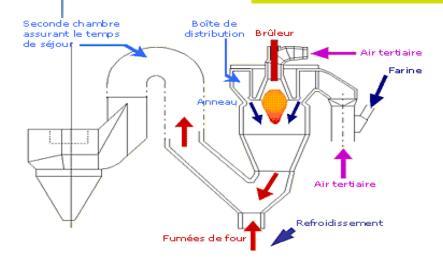
#### Product available:

❖ ARGICAL M-1200S (France)

#### **Drawbacks**

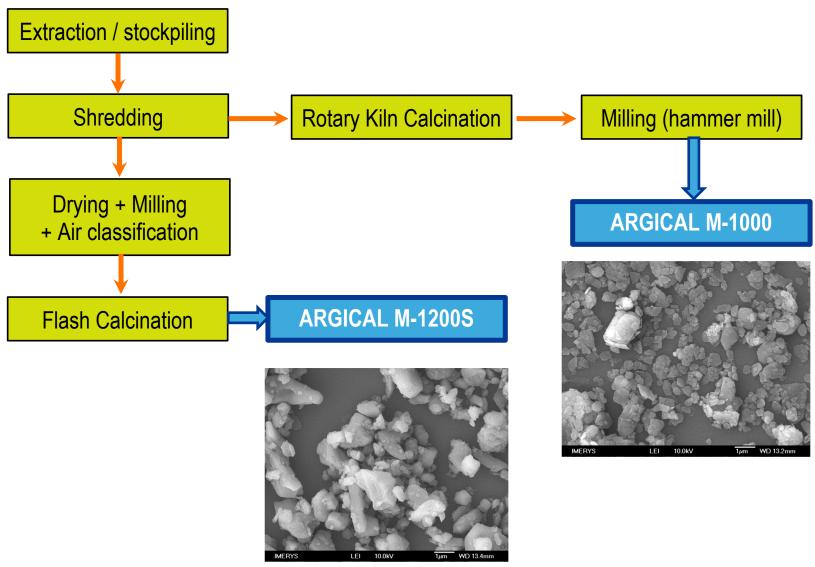
- Complex operational system.
- Important cost of investment.
- Milled material needed for feed.

Residence time: less than 1 second





## Manufacture of Metakaolin: Imerys Clérac





# **An Introduction to Metakaolins**



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## **Properties of Metakaolin: Pozzolanic Activity**

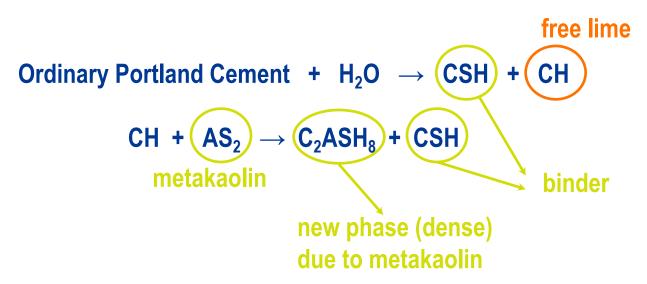
- Pozzolans are inorganic material composed mainly by rich siliceous or silicoaluminous amorphous phases.
- Pozzolans themselves possess no binder properties.
- In the presence of water, pozzolans chemically react with calcium hydroxide to produce compounds with binder properties.





## **Properties of Metakaolin: Pozzolanic Activity**

- Metakaolin is an amorphous state of kaolinite obtained by firing the mineral at a temperature between 700 and 950°C.
- It is a pozzolanic material, i.e. it reacts with lime in the presence of water.
  - ◆ In cementious materials, metakaolin reacts with the lime released by the cement during its hydratation; it also reacts with the lime added in some mortars.
  - ◆ Pozzolanic reaction:



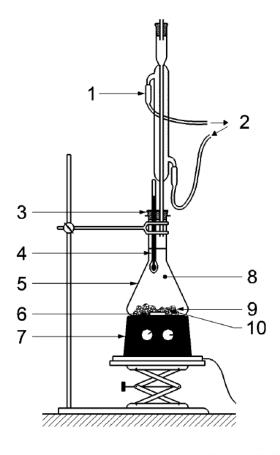


## **Properties of Metakaolin: Pozzolanic Activity**

 On a day-to-day basis, the pozzolanic activity is measured via the modified Chapelle test.

It is an indirect measurement, based on the consumption of Ca(OH)<sub>2</sub> in a saturated

water medium.



- 1: Condenser
- 2: Circulation of water
- 3: Stopper with 2 holes
- 4: Thermometre
- 5: Stainless steel erlanmeyer
- 6: Magnetic stirring bar
- 7: Heating plate with magnetic stirrer
- 8: CO<sub>2</sub>-free distilled water
- 9: CaO (2g)
- 10: Metakaolin (1 g)



# **Metakaolins from Imerys**

|                                   | MetaStar 501    | ARGICAL M-<br>1200S | ARGICAL M-<br>1000 | MK-40       |
|-----------------------------------|-----------------|---------------------|--------------------|-------------|
| Origin                            | USA             | France              | France             | Ukraine     |
| Calcination                       | Herreshoff kiln | flash kiln          | rotary kiln        | rotary kiln |
| Pozzolanic index [mg Ca(OH)2 / g] | 1400            | 1400                | 1100               | 1000        |
| Surface area<br>(BET) [m²/g]      | 14              | 23                  | 20                 | 15          |
| D50 [µm]                          | 1.0             | 1.5                 | 6.0                | 20.0        |
| Brightness                        | 85              | 72                  | 69                 | 65          |





# www.imerys.com

