



## Research paper

# Synthetic talc advances: Coming closer to nature, added value, and industrial requirements



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## ABSTRACT

Over the past 2 years, the synthetic process of talc particles has evolved considerably, leading to an inexpensive, convenient, and rapid process that is compatible with industrial requirements. In addition to facilitate the synthetic talc preparation, the evolution of the synthesis process has led to an improved crystallographic arrangement of the talc particles in both the  $c^*$  direction and (ab) plane. In the present study, the most recent process was investigated with respect to the reaction time, temperature, pressure, pH, and salt concentration to determine the optimal reaction parameters. In the geomaterial industry, X-ray diffraction is routinely used for powder material characterization; the crystallinity of our synthetic talc was evaluated by this technique through measurements of the Coherent Scattering Domain (CSD) size. A crystalline lamellarity index was defined as the ratio between the CSD size values in the (ab) plane and  $c^*$  direction. These crystallinity characteristics were used to define the quality of the synthetic talc and its suitability for potential industrial markets.

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## 1. Introduction

Talc, a layered magnesium silicate mineral with the ideal formula  $Mg_3Si_4O_{10}(OH)_2$ , is commonly used as a filler in composite materials to reduce their production costs, improve their physical and chemical properties, and provide new functionalities. It is used in numerous industrial applications (papers, paints, ceramics, cosmetics, and polymers; (Ferrage et al., 2002)) for its mechanical properties, barrier effects, and lubricating properties up to 900 °C. However, the use of this latter property in surfaces for the aeronautical sector (Martin et al., 2006, 2009) demonstrated the limitations of the particle size of natural talc (Sanchez-Soto et al., 1997). Natural talc cannot be ground homogeneously below 1  $\mu\text{m}$  without leading to amorphization of the structure. To resolve this issue and to control particle size, we turned to talc obtained from hydrothermal synthesis.

The first talc synthesis appeared in studies of equilibrium systems devoted to understanding the stability of metamorphic minerals and mineral assemblages, but this process generally required very high temperature and/or pressure (Bowen and Tuttle, 1949; Eberl et al., 1978; Johannes, 1969; Roy and Roy, 1955). Then, in an investigation of soils and sediments, new information was obtained from the study of synthetic clay single-phase specimens (Kloprogge et al., 1999; Zhang et al., 2010). In the late 1980s, Decarreau et al. (1989) used low-temperature and autogenous-pressure hydrothermal treatment

(80–240 °C) to obtain stevensite, kerolite, and talc. The starting material was a gel with a Mg/Si ratio identical to that of talc (i.e., 3/4). Decarreau et al. (1989) demonstrated that the stability of the mineral depended on the temperature of the hydrothermal synthesis and concluded that the starting temperature for talc crystallization was approximately 170 °C. However, this method led to an unstable synthetic talc, as observed in a retromorphosis experiment during which the crystallinity decreased to form kerolite.

When the replacement of natural talc by synthetic talc was envisaged in specific industrial applications, the considerations included the cost of the reactants, the reaction time, and the energy requirements. Since 2004, efforts have been focused on developing an inexpensive, stable, highly pure product of submicronic size.

The present study aims at fine-tune the most recent synthetic process and to assess the influence of several parameters, such as reaction time, temperature, pH, the addition of sodium acetate, and external pressure, on the crystallinity of talc products as assessed through X-ray diffraction (XRD) measurements. The synthetic procedure is discussed with regard to specific industrial applications.

## 2. Experimental section

Synthetic talc samples were prepared using the processes described in different patents (Dumas et al., 2012, 2013a, 2013b, 2013c; Martin et al., 2008b, 2008c). The processes are named in chronological order as P1, P2, and P3. The main steps of talc synthesis and the reactants used for each process are listed in Table 1.

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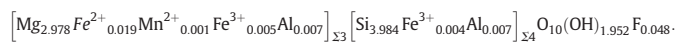
E-mail address: [angela.dumas@get.obs-mip.fr](mailto:angela.dumas@get.obs-mip.fr) (A. Dumas).

**Table 1**  
Evolution of the synthetic talc preparation process.

Year	1989	2008	2008 (P1)	2012 (P2)	2013 (P3)
	<b>Precipitation --&gt; talc precursor</b>				
Reactants		Hexahydrated magnesium chloride (MgCl <sub>2</sub> ·6H <sub>2</sub> O)	+		Magnesium acetate tetrahydrate (Mg(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O) + Pentahydrated sodium metasilicate (Na <sub>2</sub> SiO <sub>3</sub> ·5H <sub>2</sub> O) + Sodium acetate trihydrate (NaCH <sub>3</sub> COO·3H <sub>2</sub> O)
	Drying and grinding		Rinsing		
	<b>Hydrothermal treatment</b>				
Form of talc precursor additive	Powder		Powder or gel		Gel
Temperature (°C)	170 < T < 240		300 < T < 600	250 < T < 600	250 < T ≤ 350
Duration	15 days to few months	24 hours to few months	24 hours to tens of days	several hours to several days	few hours to several days
	Rinsing				
	<b>Drying and grinding</b>				
	Annealing				

### 2.1. Starting materials

The starting materials for the experiment using the most recent process (P3) were as follows: magnesium acetate tetrahydrate (Mg(CH<sub>3</sub>COO)<sub>2</sub>·4H<sub>2</sub>O), sodium metasilicate pentahydrate (Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O), sodium acetate trihydrate (NaCH<sub>3</sub>COO·3H<sub>2</sub>O), and acetic acid. All reagents were purchased from Aldrich and used without further purification. A 1 N solution of acetic acid was prepared using deionized water. As a reference material, a sample of natural talc from the Trimouns deposit in the French Pyrenees was used for comparison with the synthetic talc. Its formula is as follows (Martin et al., 1999):



### 2.2. Sample preparation

Synthetic talc samples were prepared using the new process described in a recent patent (Dumas et al., 2013a, 2013b). The preparation requires two steps: 1) the preparation of a talc precursor at room temperature with the proper Mg/Si talc ratio, obtained by coprecipitation of sodium metasilicate and magnesium acetate, and 2) hydrothermal treatment. In some attempts, the talc precursor was partially modified by pH adjustments using acetic acid, sodium hydroxide, or different salt concentrations. Synthetic parameters, such as temperature, pressure, and duration, were also modified. The temperature inside the autoclave could be precisely controlled by a thermocouple. A neutral gas, such as argon, could be added to the titanium autoclave, and the internal pressure could be controlled by a pressure gage. The parameters were modified individually according to the following reference conditions: a precursor pH of 8.8, sodium acetate (NaAc) at a concentration of 4 M, a temperature of 300 °C, a related autogenous pressure of

85 bars, and a hydrothermal treatment of 6 h. The experimental details are listed in Table 2.

### 2.3. Method

XRD patterns were recorded on an INEL CPS 120 powder diffractometer with CoKα<sub>1+2</sub> radiations between 0.334 and 107.206°2θ with a step size of 0.032 (GET, University of Toulouse). The samples were prepared using the side-loading procedure in order to minimize preferential orientations. Crystallinity evaluation in the c\* direction is based on the Coherent Scattering Domain (CSD) size estimated from the 001 and 003 reflections by the Scherrer relation (Scherrer, 1918):

$$L = \frac{K \times \lambda}{B \times \cos\theta} \quad (1)$$

where  $L$  is the CSD (Å),  $\lambda$  is the wavelength (Å),  $K$  is a constant,  $B$  is the width at half maximum (rad), and  $\theta$  is the angle of the diffraction reflection. As recommended by Brindley and Brown (1980), we used the constant  $K = 0.91$ . Coherent Scattering Domain (CSD) size in the c\* direction is an estimation of the length of the layers stacking order. Through the same relation, the dimensions of talc crystallites in the (ab) plane were also evaluated based on the 06/-33/ diffraction maxima. In the (ab) plane, the CSD size describes the expansion without any defect of the octahedral sheet. The CSD measured from the 06/-33/ diffraction maxima is an average coherency radius of the coherency ellipsoid. This geometry results from the vector product of the different bands that composed this reflection.

Uncertainties of the  $L$  values were calculated by applying the Scherrer equation for  $B + \Delta$  and  $B - \Delta$ , where  $\Delta$  is the angular step size used for the XRD recordings.

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