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Properties of an amorphous magnesium silicate synthesized by precipitation.

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Abstract

Natural talc is a very interesting filler for plastic and rubber materials and paints because it improves their properties by delivering mechanical reinforcement and barrier effects. A good dispersion in a polymer matrix involves fine particles, with a size in the order of nanometer. But at the moment the main drawback of the use of natural talc is that, with conventional milling process, particles size under one micrometer are difficult and expensive to obtain. This study objective is to develop a process of magnesium silicate synthesis made by precipitation. This step is the first in the manufacturing process of synthetic talc. The resultant product will have properties that are not possible to obtain with the milling process, in particular particle size smaller than one micrometer.

The influence of different process parameters (reactants' addition mode, reactants' concentrations, synthesis temperature and ultrasound) on product properties is studied. Synthesized product is very agglomerated whatever process parameters but some parameters would enable to decrease agglomerates size. Reactants' addition by a mixing system with ultrasound during the synthesis would be the best in order to synthesize smallest particles. Moreover synthesis temperature and initial reactants' concentrations influence the nucleation rate and so agglomeration phenomenon.

Keywords: agglomeration, crystallisation, particle, powders, precipitation

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1. Introduction

Natural talc is an interesting material that can be used as high performance filler in polymers. It improves their properties, for instance, excellent mechanical reinforcement and barrier effects are improved (Ciesielczyk, Krysztafkiewicz, Bula and Jesionowski, 2010). In plastics, talc improves tensile and transverse strength and modulus of elasticity (Tufar, 2000). In rubbers, talc is interesting because of its lubricating properties (Tufar, 2000). Talc also improves resistance to corrosion of paints. It also changes their flow properties, makes sprawl easier and reinforces hiding power (Martin, 1999). However, an homogeneous dispersion is necessary to obtain these different performances. A good dispersion in a polymer matrix needs, in an ideal case, nano-sized particles (Martin, Ferret, Lèbre, Petit, Grauby, Bonino, Arseguel and Decarreau, 2006). However, it is difficult to obtain particles smaller than one micrometer using conventional milling processes. An alternative method to milling is the chemical synthesis of talc. Two steps are necessary. The first step consists of silicate magnesium precipitation with the production of an amorphous solid. An second step is necessary to convert the amorphous solid into a crystalline lamellar nano-sized particles with a talc-like structure. Amorphous or crystalline magnesium silicate particles can be used as filler particles.

The second step of the process of synthetic talc has been the subject of several investigations by Lèbre (2007). But, except works of Ciesielczyk, Krysztafkiewicz and Jesionowski (2005, 2007), Ciesielczyk et al. (2010), few investigations have been held about the precipitation. The aim of this work is to study the influence of precipitation parameters on product properties.

Though some process parameters, such as reactants' concentrations, temperature or use of a premixing system or ultrasound present an influence on properties of a precipitated product, in particular on particles size distributions(Mersmann, 1999). According to Dodds, Espitalier, Louisnard, Grossier, David, Hassoun, Baillon, Gatumel and Lyczko (2007), ultrasound used during the synthesis also influence particle size. It has been shown that $BaSO_4$, K_2SO_4, TiO_2 and sucrose crystals are smaller when crystallisation or precipitation is made with ultrasound. In this work, ultrasound influence on particles size distributions is studied.

Synthesized particles size distributions and agglomeration phenomenon is the most important property observed in this study.

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2. Experimental

2.1. Materials

The amorphous magnesium silicate is synthesized by a precipitation between a sodium metasilicate solution (Na_2OSiO_2) and an acid magnesium chloride solution ($MgCl_2$), according to the following equation 1 (Martin et al., 2006):

$$4Na_2OSiO_2, 5H_2O + 3MgCl_2, 6H_2O + 2HCl \rightarrow Si_4Mg_3O_{11}n(H_2O) + 8NaCl + H_2O \quad (1)$$

Reactants are aqueous solutions of sodium metasilicate (purity 100%), called reactant 1, and of magnesium chloride (purity 97%), called reactant 2 (supplier Prolabo). Molar ratio Mg/Si is always 0.75, corresponding to that of natural talc. In order to maintain stoichiometry of reaction, hydrochloric acid 1M is added in aqueous solution of magnesium chloride (supplier Prolabo). Demineralised water is used. At the end of the synthesis, a part of the solid is dried at 550°C during 5 hours. The solid weight loss permits to estimate the value of n. It's ranged between 4.6 and 4.85. The influence of reactants' addition mode, reactants' concentrations ($C_{MgCl_2,6H_2O}$ = 0.81, 1.49 and 1.88 mol.L^{-1}; $C_{Na_2SiO_3,5H_2O}$ = 0.71, 1.43 and 1.97 mol.L^{-1}), temperature (20, 40 and 50°C) and ultrasound in the Y-mixing system on product properties is studied.

2.2. Procedures and Methods

2.2.1. Synthesis of magnesium silicate

This synthesis is made in a 1L vessel with four baffles, at constant temperature and stirrer rate of 600 rpm. The mixing device is an PTFE helix propeller with four squared blades. Its diameter is about 4 cm. Three addition modes have been tested. The first mode is the slow addition, drop by drop, of one reactant into the other (Fig. 1a). The second mode is the fast addition of one reactant into the other. This mode is not depicted in Fig. 1 because materials are the same as slow addition. The only difference is that magnesium chloride solution is added instantaneously instead of drop by drop. In the third mode, reactants are mixed in a Y-mixing system ($H \approx 6$ cm, $D \approx 4$ cm, $V \approx 42$ mL)(Fig. 1b). Peristaltic pumps are used in order to feed solutions into the mixing system. Rates and speeds of flow are respectively 125.25 g.min^{-1} and 0.30 m.s^{-1} for sodium metasilicate solution and 81.30 g.min^{-1} and 0.19 m.s^{-1} for magnesium chloride solution. Mixing occurs inside the mixing system at the exit of pipes which come from pumps. At the exit

of the mixing system, the suspension is poured in the vessel which is initially filled with 100 mL of demineralised water. Ultrasound can be used in the Y-mixing system (f=20 kHz).

In the Y-mixing system, the Reynolds number $\mathrm{Re_Y}$ is given by the following equation (Eq. 2):

$$Re_{\mathrm{Y}} = \frac{\rho \mathrm{Lu}}{\mu} = 1\ 184 \tag{2}$$

with ρ, the density of the suspension ($\mathrm{kg.m^{-3}}$), L, the Y-mixing system diameter (m), u, the flow speed ($\mathrm{m.s^{-1}}$) and μ, the viscosity of the suspension ($\mathrm{kg.m^{-1}.s^{-1}}$).

This value of the Reynolds number indicates an intermediate range between laminar and turbulent flows. In the vessel, the Reynolds number of mixing $\mathrm{Re_v}$ is given by the following equation (Eq. 3):

$$Re_{\mathrm{v}} = \frac{\rho \mathrm{ND}^2}{\mu} = 21\ 876 \tag{3}$$

with ρ, the density of the suspension ($\mathrm{kg.m^{-3}}$), N, the revolution speed of the mixing device ($\mathrm{r.s^{-1}}$), D, the mixing device diameter (m) and μ, the viscosity of the suspension ($\mathrm{kg.m^{-1}.s^{-1}}$).

This value indicates a turbulent range. The Reynolds number in the vessel enables to determine the power number $\mathrm{N_p}$ for the used mixing device ($\mathrm{N_p}$ is 1 in this study) and then the energy dissipation ϵ (Eq. 4):

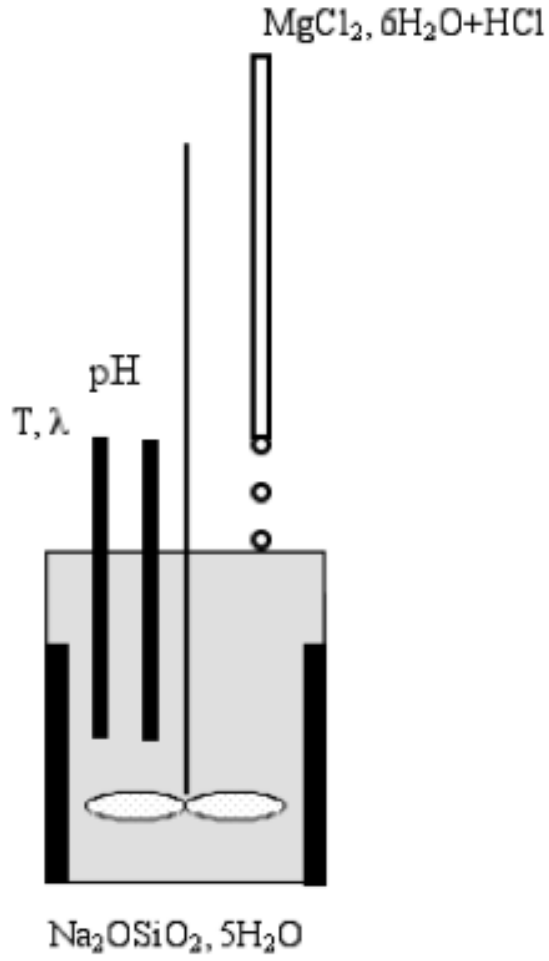
$$\epsilon = \frac{N_{\mathrm{p}} N^3 D^5}{V} = 0.18\ \mathrm{W.kg^{-1}} \tag{4}$$

with V, the vessel volume ($\mathrm{m^3}$).

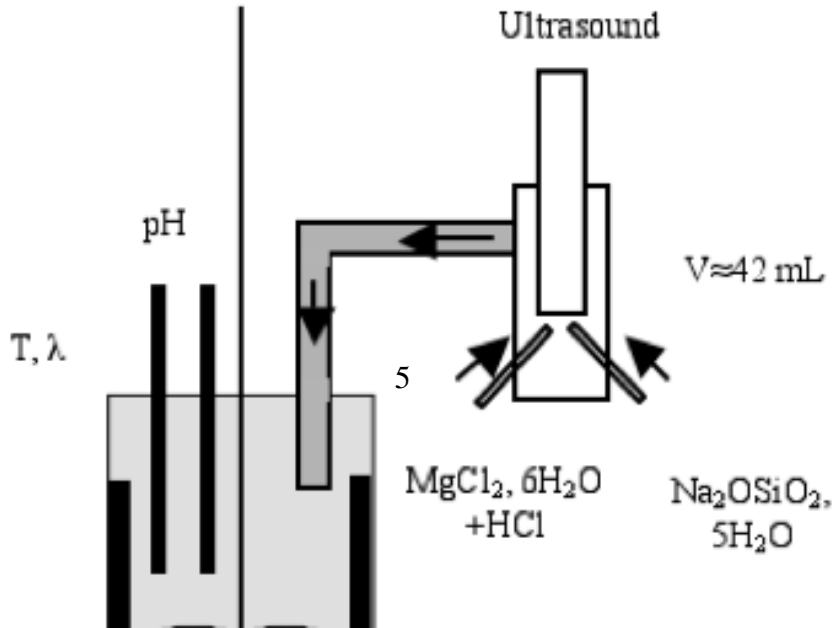
When trials are made at a higher temperature than the room temperature (40 or 50°C) reactants are heated with hotplates. The vessel is heated at 40 or 50°C thanks to a constant-temperature bath.

During the synthesis, the conductivity and the temperature of the solution are measured. Getting the stability of the conductivity enables to determine the end of the reaction. The conductivity of a solution is influenced by the temperature. So conductivities measured at different temperatures must be corrected by a temperature coefficient in order to be compared. The temperature coefficient θ (%/°C) is calculated by the following equation (Eq. 5):

$$\theta = \frac{(\lambda_{\mathrm{T2}} - \lambda_{\mathrm{T1}}) * 100}{(T_2 - T_1) * \lambda_{\mathrm{T1}}} \tag{5}$$


MgCl2, 6H2O+HCl
pH
T, λ
Na2OSiO2, 5H2O

(a)


Ultrasound
pH
T, λ
V≈42 mL
MgCl2, 6H2O
+HCl
Na2OSiO2,
5H2O

with λ_{T2}, the conductivity measured at the temperature T_2, λ_{T1}, the conductivity measured at the temperature T_1 which is close to the reference temperature (20 °C). Conductivities unit is S.cm^{-1} and temperatures unit is °C.

In this work, the temperature coefficient θhas been measured with a solution containing ... for a temperature range of 20-40 °C . A value of 2.1 %/°C has been obtained.

At the end of the synthesis, the suspension is filtered with a Büchner-membrane system. The cake is then washed four times with water, so salt formed during the synthesis is eliminated. The product is then dried at 100°C during 12 hours. At the end of the drying step, the product is very agglomerated that's why it's ground by a laboratory impact mill (coffee mill).

2.2.2. Properties of synthetic magnesium silicate

Suspension and synthesized powders are submitted for physical chemistry analysis.

Samples are taken of the suspension in order to be analysed by dynamic light scattering technique using Malvern Mastersizer 2000. This enables particle size distribution to be followed during the synthesis. Suspension is diluted with water in the granulometer. Ultrasound can be applied during the size measurement. Figure 2 shows the influence of ultrasound applied during the size analysis on particle size distribution. After ultrasound use, size distribution is shifted towards fine particles. It must be due to ultrasound which cause disaggregation of the solid. Over two minutes of ultrasound, particles size distribution doesn't change any more. So every sample is subjected to ultrasound during two minutes.

In order to define surface morphology and agglomeration state of particles, dried products are examined by scanning electron microscopy (SEM-FEG, Philips XL 30).

Density of dried products is measured using a Micrometrics AccuPyc 1330 helium pycnometer.

BET surface area of dried products is estimated using a Micrometrics ASAP 2010 K2 apparatus and getting nitrogen adsorption/desorption isotherms. Mesoporous volume is calculated by using the Barret, Joyner and Halenda method (B.J.H. method) on the desorption field of the adsorption/desorption isotherm. Micro-porous volume is calculated by using the Horvath-Kawazoe method (H. –K. method) with split pore geometry on the adsorption field of the adsorption/desorption isotherm.

Analysis by X-Ray diffraction are also made on dried products using the diffractometer X'Pert Philips Pan Analytical with CuKα radiation source. Mea-

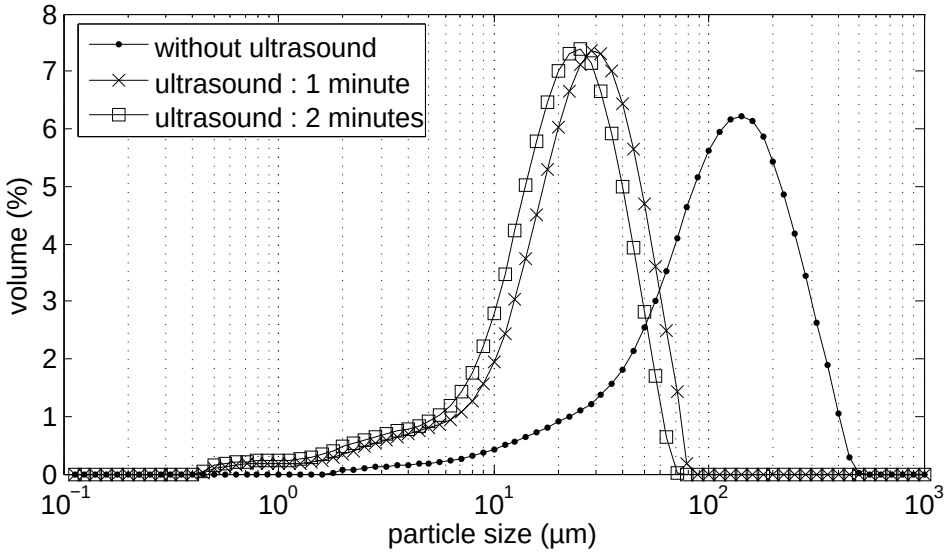


Figure 2: Influence of ultrasound applied during the size analysis on particle size distribution (addition mode: slow addition, $C_{MgCl_2,6H_2O} = 1.49$ mol.L^{-1}, T=20°C, samples taken of after 210 minutes).

surement and duration steps are respectively 2θ=0.0334° and 50 seconds.

2.2.3. Amorphous magnesium silicate solubility

Some trials on magnesium silicate solubility are made at different temperatures. Solubility is measured in water for a constant concentration of NaCl equal to 0.63 mol.L^{-1}. ~~The concentration of NaCl is the same as concentration in the vessel during the synthesis (0.63 mol.L^{-1}).~~ Dried and milled magnesium silicate is introduced in the aqueous solution kept at ~~some~~ six constant temperatures (20, 30, 35, 40, 50 and 60°C plus ou moins ??). Conductivity is measured as a function of time, while suspension is agitated (Fig. 3).

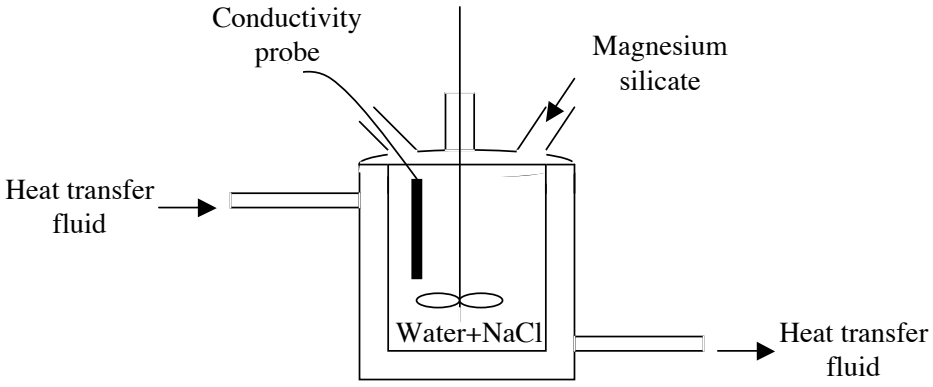


Figure 3: Experimental scheme of solubility trials.

3. Results and discussion

3.1. Amorphous magnesium silicate solubility

Figure 4 shows the evolution of suspension conductivity as function of time at constant temperature. After two hours, the conductivity attains a plateau. Stability of conductivity reveals equilibrium state of the suspension. The value at the plateau increases with the temperature. So amorphous magnesium silicate solubility seems to increase when temperature increases (Fig. 4).

3.2. Evolution of the conductivity during synthesis

The conductivity of the suspension is measured in the vessel during the synthesis. The evolution of the conductivity is influenced by the addition mode (Fig. 5).

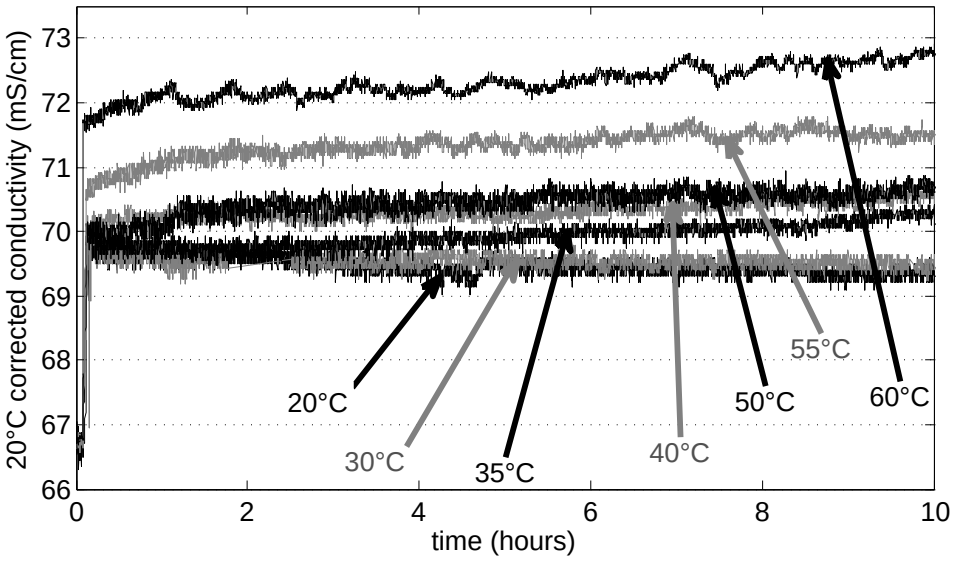


Figure 4: Evolution of conductivity as a function of time and temperature for a constant concentration of NaCl equal to 0.63 mol.L^{-1}.

In the process with the slow addition, the conductivity firstly slowly decreases because of the solid formation by addition of magnesium chloride solution in the vessel. The conductivity is then stable before increasing at the end of the addition. This increase is not explained yet. In the process with the fast addition, the conductivity fastly decreases because of the solid formation by addition of magnesium chloride solution in the vessel. The conductivity is then stable. The final value is the same as in the process with the slow addition. In the process with the mixing system, the conductivity firstly fastly increases because the vessel is initially filled with water. The conductivity is then stable because the solid is formed before being poured in the vessel. In this case, the final conductivity is lower than slow and fast addition. The desupersaturation seems to be more important in the case of reactant pre-mixing.

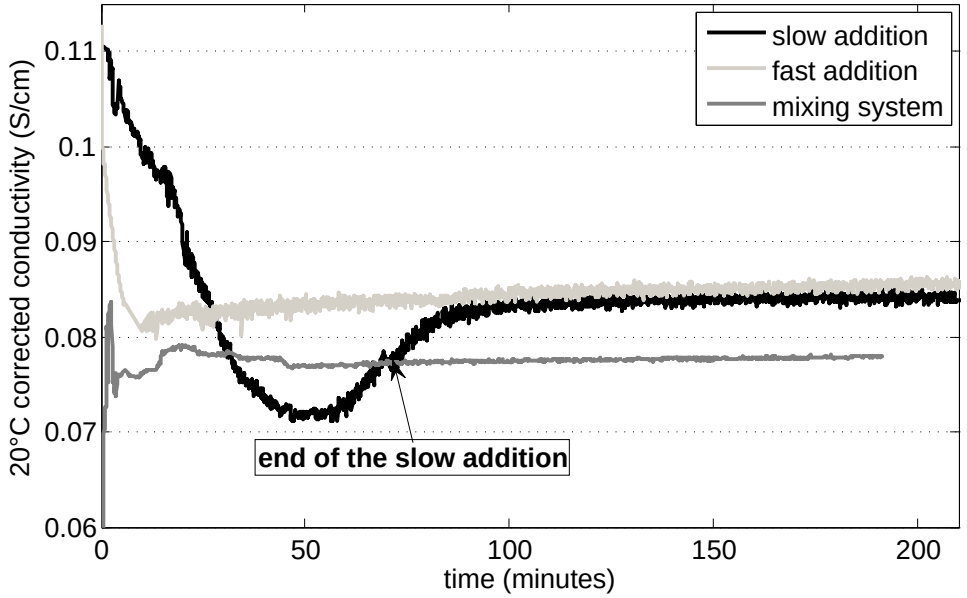


Figure 5: Evolution of the conductivity as a function of time and addition mode ($C_{MgCl_2,6H_2O}$ = 1.49 mol.L^{-1}, T=20°C).

The conductivity of the suspension at the end of the synthesis, is also influenced by the temperature and the NaCl concentration (Fig. 6). When NaCl concentration is ~~fixed~~ constant, the final corrected conductivity increases with the temperature in agreement with the evolution of solubility as function of temperature. When the temperature is ~~fixed~~ constant, the final conductivity increases with

the NaCl concentration. ~~It's explained by the increase of dissolved ions when NaCl concentration increases.~~ From these different results on synthesis and solubility measurements, the solubility of amorphous magnesium silicate increases with temperature and NaCl concentration or ionic strength.

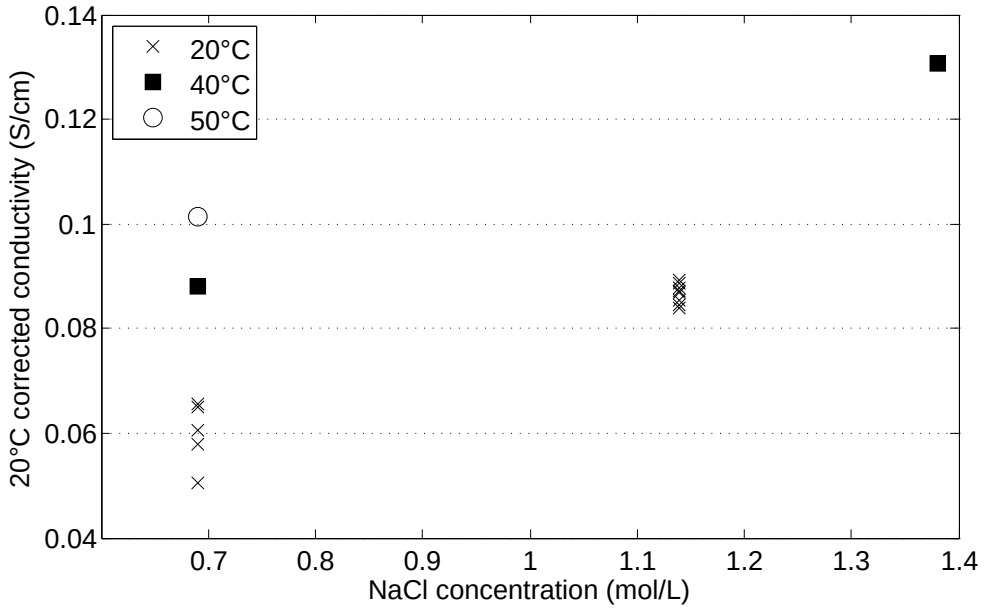


Figure 6: Conductivity at the end of synthesis as a function of temperature and NaCl concentration.

3.3. Properties of synthetic magnesium silicate

3.3.1. Amorphous state of the powder

Diffractograms get by X-Ray diffraction analyses show that all synthesized magnesium silicate solids are amorphous. The diffractogram (Fig. 7) doesn't present any real peak, only a large initiation of a peak at 2θ=46.5°. But it can't be exploited because of its low intensity.

Whatever studied process parameters, product ~~get after the step of~~ obtained by precipitation is always amorphous. These results are in compliance with works of Ciesielczyk et al. (2005, 2007). They have showed that precipitated magnesium silicate is amorphous and this form is independent of the precipitation temperature and addition mode.

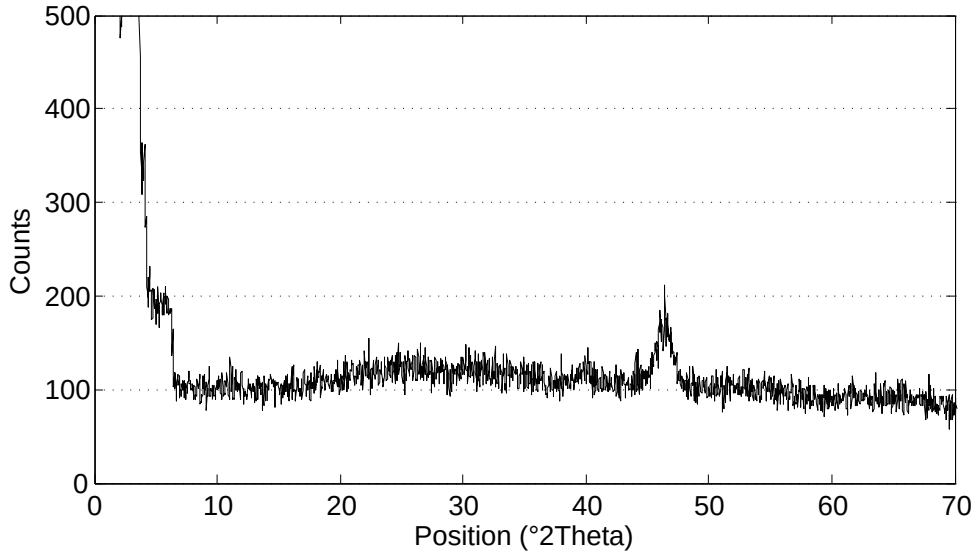


Figure 7: Diffractogram of a product (addition mode: parallel with mixing system, $C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1}, T=20°C) get by X-Ray diffraction at 0.154 nm (Cu) (measurement step 2θ= 0.0334°; duration step = 50 seconds).

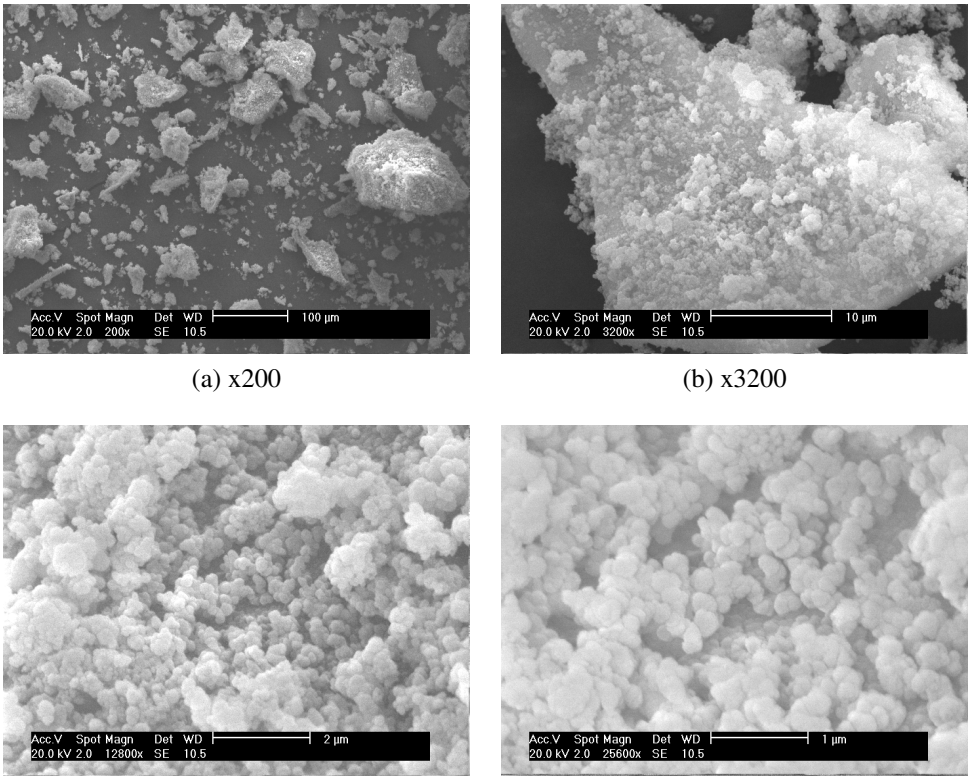
3.3.2. Agglomeration state of the powder

The dry solid is composed of fine particles strongly agglomerated (Fig. 8). Amorphous product seems to be made up of two kinds of agglomerates. Secondary agglomerates (Fig. 8a and 8b) are made up of primary agglomerates (Fig. 8c) which are made up of primary spherical particles (Fig. 8d). This kind of structure is observed whatever process parameters.

The Table 1 summarizes suspension and solid properties (corrected conductivity at the end of synthesis λ_s , d_{50}, d_{32}, density, specific surface area) as a function of operating conditions.

The product density seems to be independent of studied operating conditions: 2.195±0.036 g.cm^{-1}.

Except trial 1, the specific surface area varies conversely to the diameters d_{50} and d_{32}: when the diameter increases, the specific surface area decreases. The result of the trial 1 is different but it's the only specific surface area measured with the slow addition. This difference might be due to the addition mode. Maybe with the slow addition the magnesium silicate structure is formed differently than with the mixing system. More analysis would be necessary on products synthesized with the slow addition in order to conclude.



(a) x200 (b) x3200

(c) x12800 (d) x25600

Figure 8: SEM analysis of products synthesized by slow addition, $C_{MgCl_2,6H_2O} = 1.49$ mol.L^{-1}, T=20°C.

Trial	Addition mode	Temperature (°C)	$[MgCl_2, 6H_2O]$ (mol.L^{-1})	$[Na_2SiO_3, 5H_2O]$ (mol.L^{-1})	λ_f (mS.cm^{-1})	d_{50} (μm)	d_{32} (μm)	Density (g.cm^{-3})	Sp su a (m
1	slow	20	1.49	1.43	83.8	20.9	9.9	2.209	
2	fast	20	1.49	1.43	86.4	26.1	13.1	2.206	
3	mixing system +ultrasound	20	1.49	1.43	77.9	19.2	6.5	2.203	
4	mixing system (no ultrasound)	20	0.81	0.71	58.7	11.2	5.7	2.131	
5	mixing system +ultrasound	20	0.81	0.71	64.2	4.2	2.3	2.147	
6	mixing system +ultrasound	40	0.81	0.71	63,7	4.4	2.7	2.222	
7	mixing system +ultrasound	40	1.97	1.88	94.6	17.3	7.1	2.222	
8	mixing system +ultrasound	50	0.81	0.71	64.3	5.9	2.8	2.220	

Table 1: Physical properties of synthetic magnesium silicates.

Synthesized magnesium silicate has micro-pores: gas is absorbed at very low relative pressures (Fig. 9). The mean diameter of micro-pore is about 0.65 nm and the micro-porous volume is 0.10 $m^3.g^{-1}$. The product presents only an inter primary agglomerates meso-porosity. Meso-pores might be inside secondary agglomerates, between primary agglomerates whereas micro-pores might be inside primary agglomerates, between primary particles. The meso-pore diameter is 23 nm and the meso-porous volume is 0.46 $m^3.g^{-1}$. The hysteresis observed is probably due to this inter-granular porosity.

3.4. Influence of reactants' addition mode

Particle size distributions (Fig. 10) show there is an influence of reactants' addition mode. The three particle size distributions have the same width (700 nm-80 μm) but with the addition by mixing system, particle size distribution is composed of smaller particles under 7 μm. With the fast addition, particle size distribution shows less agglomerates under 10 μm but more agglomerates between 10 and 100 μm. These differences may be due to the micromixing effect.

SEM analysis (Fig. 11) confirm these observations: product synthesized with the mixing system and ultrasound is made up of smaller particles.

3.5. Influence of ultrasound during the synthesis

At low concentrations ($C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1}), with ultrasound used during the synthesis made by parallel reactants' addition with the mixing system,

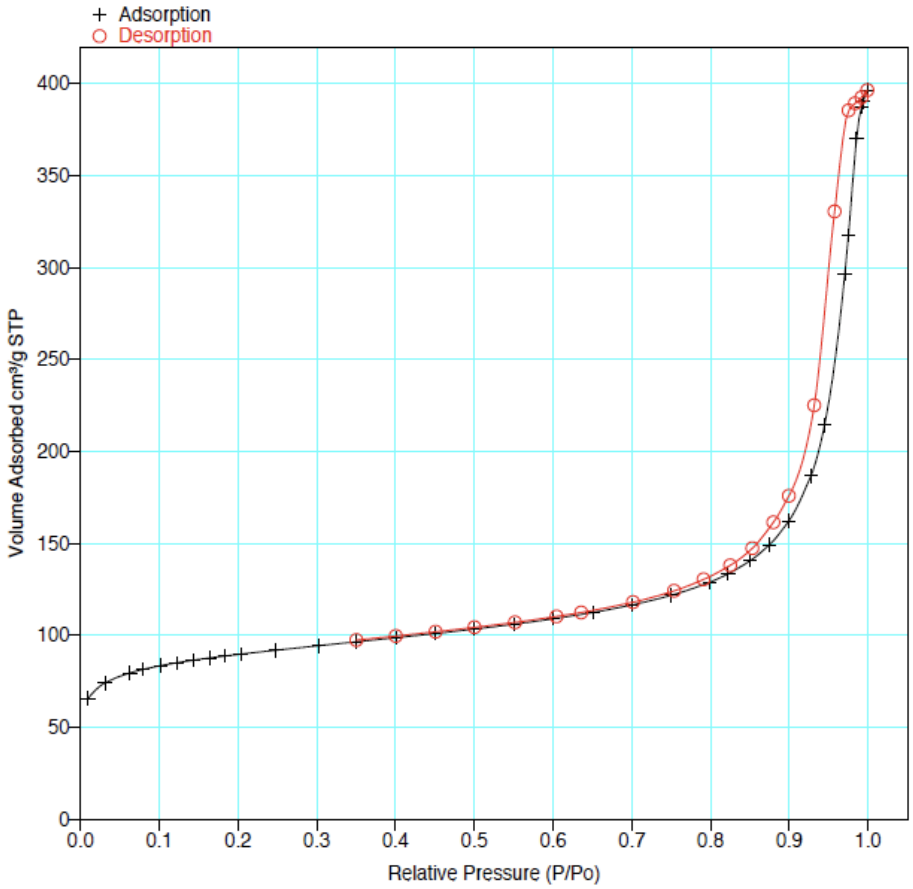


Figure 9: Adsorption/desorption isotherm of a magnesium silicate (parallel reactants' addition with mixing system and ultrasound are used, $C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1}, T=20°C).

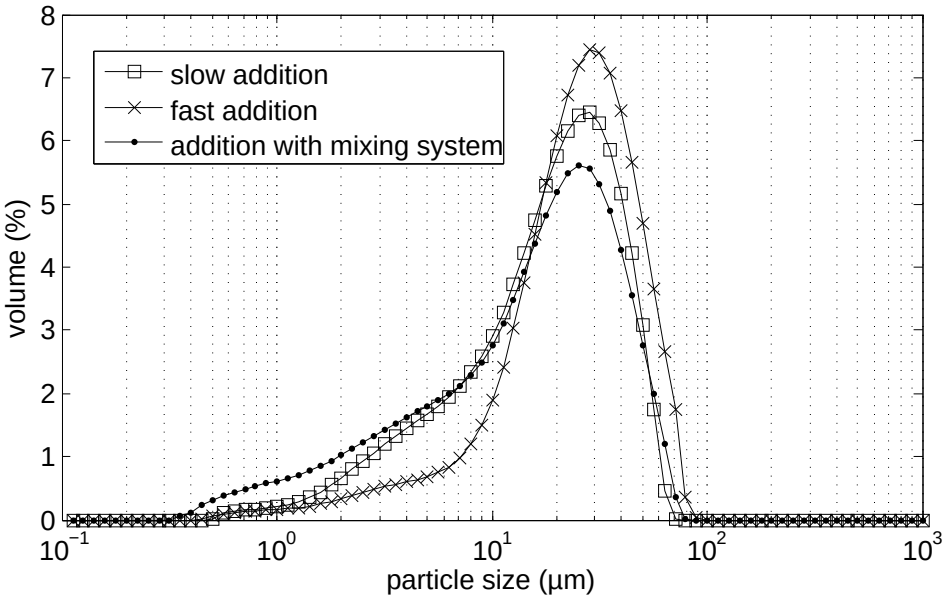


Figure 10: Particle size distributions as a function of addition modes ($C_{MgCl_2,6H_2O}$ = 1.49 mol.L^{-1}, T=20°C, samples taken of after 90 minutes).

smaller particles are observed (Fig. 12). Particle size distributions are very different. Particle sizes are smaller than 20 μm with ultrasound as against 100 μm without ultrasound in the mixing system.

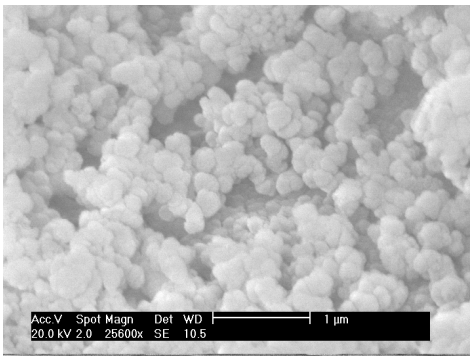
Ultrasound would reduce particles size, prevent agglomerates from forming and would break flocs already formed during the mixing of reactants. Same results on size reduction have been found with the synthesis of $BaSO_4$, K_2SO_4, TiO_2 and sucrose particles (Dodds et al., 2007). In these works, ultrasound treatment produces a decrease of particle size in comparison with the same physical or chemical process without ultrasound.

With ultrasound in the mixing system, the specific surface area of the product is 350 m^2.g^{-1} as against 257 m^2.g^{-1} without ultrasound (Table 1).

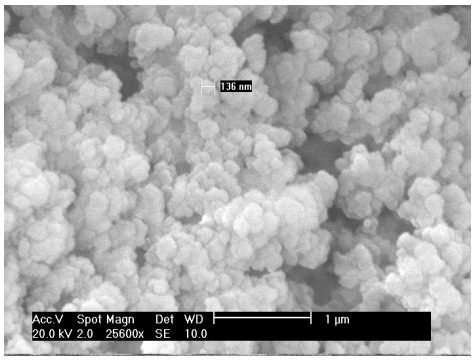
3.6. Influence of reactants' concentrations

Particle size distribution is very influenced by initial reactants' concentrations. At high concentrations, in the same other process conditions, distribution is clearly shifted towards large sizes (Fig. 13).

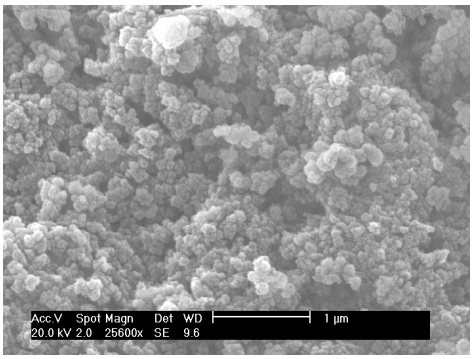
At high concentrations (1.49 mol.L^{-1}), the particle size distribution is almost the same as distribution obtained with no mixing system fast addition).



(a) x25600



(b) x25600



(c) x25600

Figure 11: SEM analysis of products synthesized by slow addition (a), fast addition (b) and with the mixing system and ultrasound (c) ($C_{MgCl_2,6H_2O}$ = 1.49 mol.L^{-1}, T=20°C).

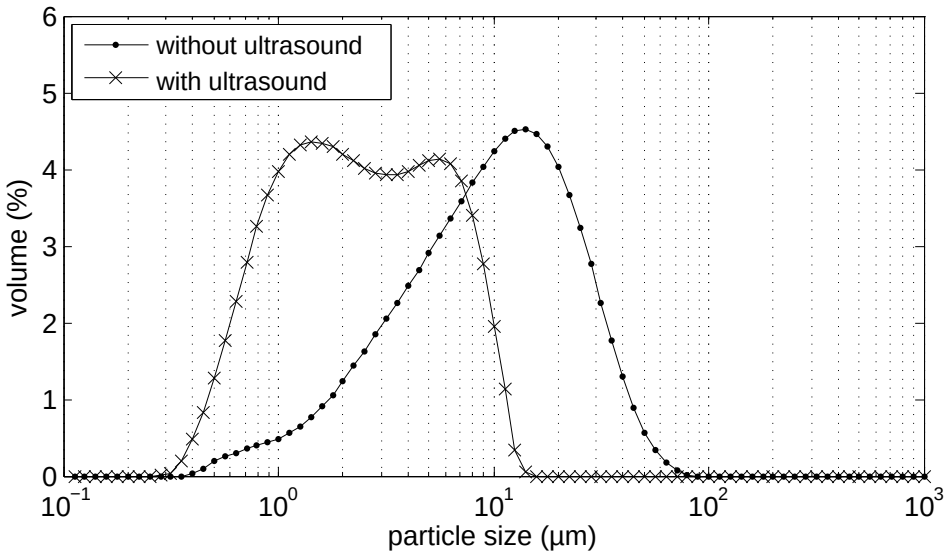


Figure 12: Particle size distributions as a function of ultrasound used during the synthesis. Parallel reactants' addition with mixing system is used ($C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1}, T=20°C, samples taken of after 90 minutes).

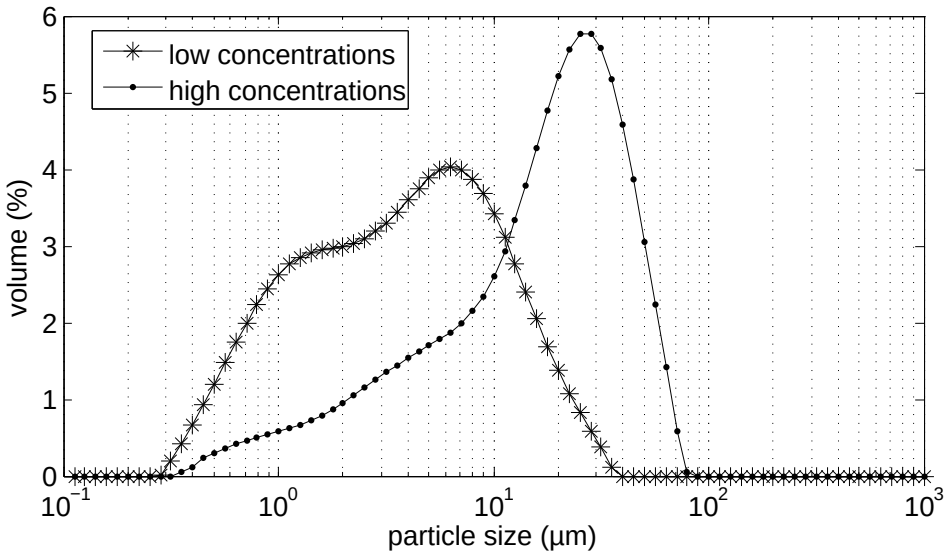


Figure 13: Particle size distributions as a function of reactants' concentrations. Parallel reactants' addition with mixing system and ultrasound are used ($C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1} and 1.49 mol.L^{-1}, T=20°C, samples taken of after 210 minutes).

The same tendency is observed at 40°C (Fig. 14).

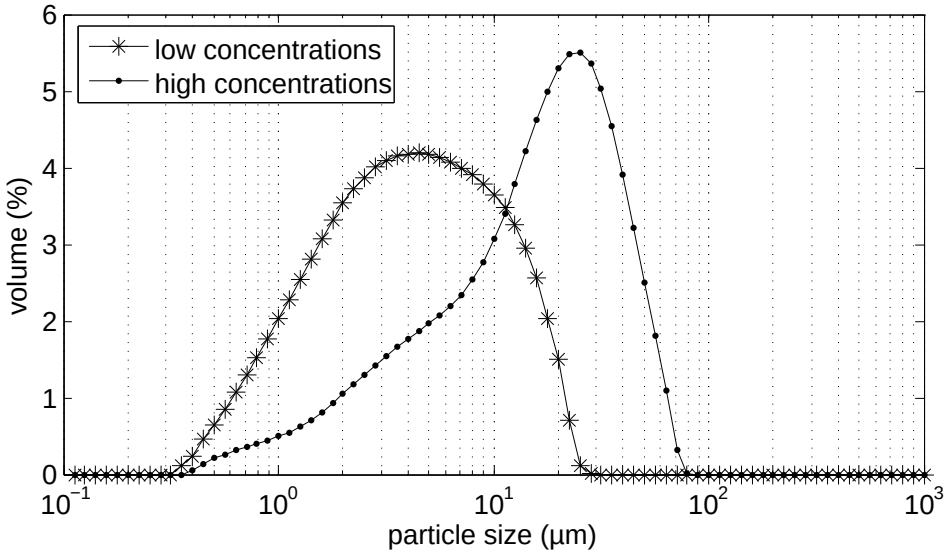


Figure 14: Particle size distributions as a function of reactants' concentrations. Parallel reactants' addition with mixing system and ultrasound are used ($C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1} and 1.88 mol.L^{-1}, T=40°C, samples taken of after 210 minutes).

These results are explained by the increase of supersaturation ratio at high concentrations. Nucleation rate also increases so more particles are synthesized. The increase of particles number causes the increase of agglomeration phenomenon. That's why distribution is shifted towards large particles at high concentrations.

Therefore, according to Zauner and Jones (2000), by increasing concentrations and so supersaturation, primary particles are smaller. This result has been confirmed by Baldyga, Makowski and Orciuch (2007). So increasing initial concentrations would enable to synthesize more smaller particles but to increase agglomeration too.

When initial concentrations are higher, the specific surface area of the product is lower: 165 m^2.g^{-1} with $C_{MgCl_2,6H_2O}$ = 1.49 mol.L^{-1} as against 350 m^2.g^{-1} with $C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1} (Table 1).

3.7. Influence of synthesis temperature

Synthesis temperature influence on particle size is studied. Particle size distributions are very similar (Fig. 15). The temperature doesn't influence the particle

size distributions. When temperature increases, specific surface area slightly decreases : 335 $m^2.g^{-1}$ at 50°C as against 350 $m^2.g^{-1}$ at 20°C (Table 1).

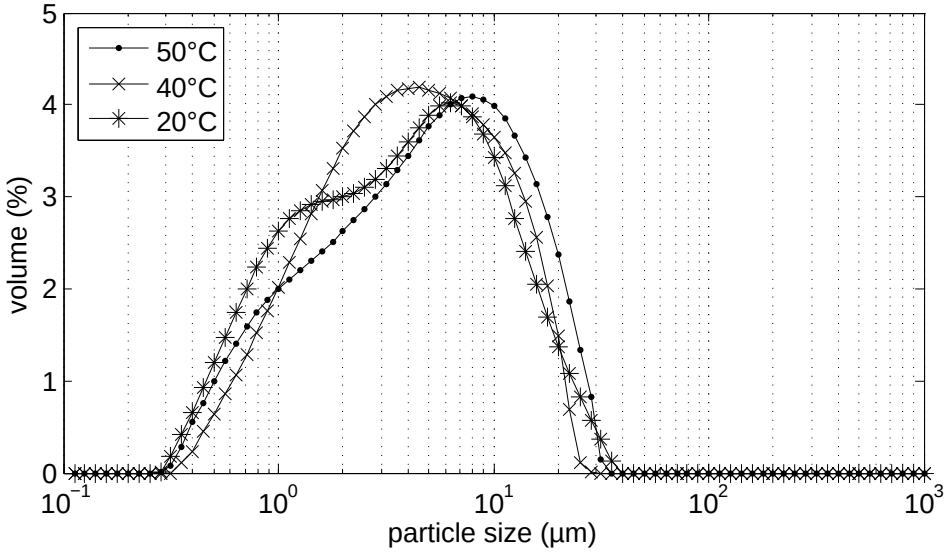


Figure 15: Particle size distributions as a function of synthesis temperature. Parallel reactants' addition with mixing system and ultrasound are used ($C_{MgCl_2,6H_2O}$ = 0.81 mol.L^{-1}, samples taken of after 210 minutes).

4. Conclusions

Studied process parameters (reactant concentration, temperature, mixing mode) wouldn't influence cristallinity rate of synthesized product, it's amorphous in all cases. It's also very agglomerated whatever process parameters. Therefore some parameters would enable to decrease agglomerates sizes. In order to synthesize smallest agglomerates and particles, reactants' addition by the mixing system with ultrasound used during the synthesis would be the most appropriate. Median agglomerates size obtained with these conditions is about 4 μm. It's bigger than the objective size but it's agglomerates size and not primary particles size. SEM analysis let think that primary particles size is about 100 nm. ~~(~~Synthesis temperaturew) Mixing mode and initial reactants' concentrations influence agglomeration phenomenon by means of the nucleation. High initial concentrations and premixing would enable to synthesize more particles and so increase agglomeration phenomenon.

A perspective of this study is to be able to easy disaggregate product in the polymer matrix in order to obtain nanosized primary particles well-dispersed in the polymer.

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