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DECARBONATION AND ATTRITION OF CALCITE IN A PLASMA SPOUTED BED REACTOR

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Les énergies mécanique et thermique de concassage et calcination sont les postes de consommation énergétique les plus importants de l'industrie du ciment. On présente dans cet article des résultats expérimentaux concernant la fragmentation et la décarbonatation de CaCO_3 dans un lit soufflé par plasma. Le paramètre essentiel est l'enthalpie spécifique du plasma : le domaine d'enthalpie étudié est compris entre 3 et 20 kWh.m^{-3} . Les évolutions de la vitesse d'attrition, de décomposition et de la distribution granulométrique du solide sont analysées.

ABSTRACT.

The mechanical power and the thermal energy for the processing of calcite are the main part of the energy consumption in cement industry. Experimental results about particle size reduction and calcination of CaCO_3 in a plasma spouted bed reactor are presented in this paper. The main parameter seems to be the specific enthalpy of the plasma jet, it ranges between 3 kWh.m^{-3} and 20 kWh.m^{-3} . The variations of the attrition rate, decomposition rate and particle size distribution are discussed.

1. INTRODUCTION.

The breaking and decarbonation of calcite are important operations of cement process. The use of thermal plasma should be a promising way of treatment if both operations should be done at the same time. In order to obtain this result we used a plasma spouted bed reactor because of its high plasma particulates heat transfer efficiency /1/.

Decarbonation of limestone in a plasma fluidized bed was studied by /2/. In the latter system, fluidized particles were heated by a plasma torch located at the wall of the column, as a consequence fluidization and heating are processed separately. The experimental data prove that :

- the reaction is controlled by external mass transfer through CO_2 gas film,
- the attrition rate is significant (the rate was not quantified) and depends on the gas composition. Particle breaking is larger in N_2 plasma than in Ar plasma.

The data presented by /3/ are related to the behavior of corundum particles in a plasma spouted bed. A large decrease of the particle diameter is observed, for example, the mean diameter decreases from 750 μm to 500 μm after a 10 minutes test in a 28 kW Ar/ N_2 plasma. The influence of the operating parameters on the attrition rate was not studied.

Concerning the selection of the operating conditions for the thermal treatment of a given solid in a plasma spouted bed reactor very few informations are available. The data from /4/ indicate a large variation of the maximum spoutable depth with particle diameter and the observation of hydrodynamical instability with small diameter particulates.

This paper deals with the analysis of the variations of both decomposition and attrition rates of calcite with plasma operating conditions in a spouted bed. The main objective is the achievement of maximum values of both rates.

2. EXPERIMENTAL METHODS.

Experimental set up. The main part of the experimental set up are presented on figure 1. The refractory column is 85 mm i.d. and angle of the cone is 60°. The fines flowing out with the gas are collected by the cyclone and samples are taken using a stainless steel tube connected with a vacuum pump. Four type K thermocouples are used in order to measure the temperature distribution inside the bed. In addition heat balances over the plasma torch and the metallic support are performed by means of flowmeters and thermocouples. The data are recorded by a data acquisition system.

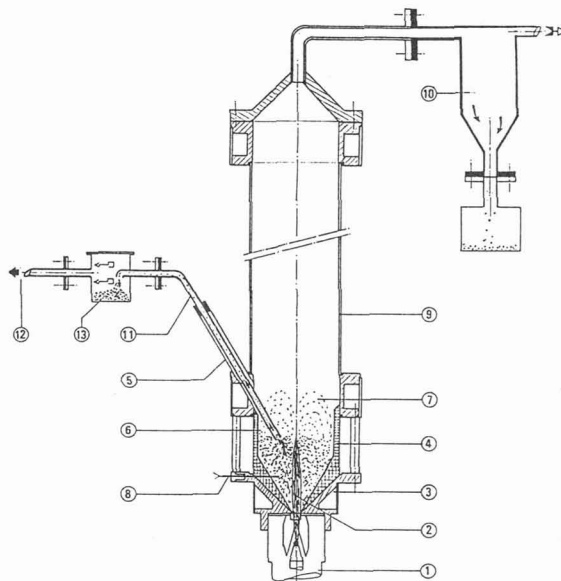


Fig. 1 - Experimental set up

- | | |
|----------------------------------|------------------|
| 1. Plasma generator | 7. Fountain |
| 2. Plasma jet | 8. Thermocouples |
| 3. Conical water - cooled vessel | 9. Silica tube |
| 4. Refractory insulation | 10. Cyclone |
| 5. Particle inlet | 11. Sample gauge |
| 6. Annulus (dense bed) | 12. Vacuum pump |
| 13. Collector. | |

Experimental procedure. Results are obtained on the basis of batch operations as follows : argon plasma is started in the empty column (typical gas flow rate of about 10 l.mn^{-1}) then a given weight (400 g) of calcite is introduced inside the reactor during about one minute. Electric conditions are chosen in order to reduce the temperature elevation during this first period. The temperature at the end never exceed 300°C . After this preliminary phase nitrogen is used instead of argon. This operation is defined as the origin for time ($t = 0$).

For each test, attrition rate (R_a) and calcination rate (X) as a function of time are measured. These data are obtained on the basis of the analysis of samples collected inside the cyclone and in the dense bed.

Definition of the rates. The attrition rate is the relative weight (M_f) of particles of diameter less than $d_{p \text{ min}}$ generated during a given duration (the selected value of $d_{p \text{ min}}$ is $80 \mu\text{m}$). Between t_{n-1} and t_n the attrition rate is defined by :

$$R_a(t_n) = \frac{M_f(t_n) - M_f(t_{n-1})}{t_n - t_{n-1}} \quad (1)$$

The total amount of fine (m^t_f) is due to the contribution of the particles collected in the cyclone (m^c_f) and in the reactor (m^R_f). Let $m_R(t_n)$, $m_s(t_n)$ be respectively the weight of solid particles in the reactor and the weight of the sample, at time t_n ; the total amount of fine in the reactor (m^{*R}_f) may be expressed as :

$$m^{*R}_f(t_n) = m^R_f [m_R/m_s] (t_n) \quad (2)$$

Finally, $M_f(t_n)$ is defined as follows :

$$M_f(t_n) = \frac{m^t_f(t_n)}{m_R(t_n)} \quad (3)$$

The developed expression is :

$$M_f(t_n) = \frac{m^{*R}_f(t_n) + \sum_{i=1}^n m^c_f(t_i)}{m_R(t_0) - \left[\sum_{i=1}^n [m^c_f(t_{i-1}) + m^s_f(t_{i-1})] \right]} \quad (4)$$

Concerning the conversion rate, X , the data were obtained by complete calcination of the sample in a 950°C furnace during 2 hours.

The calcite particles. Two particle diameters have been tested : mean diameters $111 \mu\text{m}$ and $172 \mu\text{m}$. The initial particle size distribution of the calcite is shown on figure 2. Before experiments the particles of diameter less than $d_{p \text{ min}} = 80 \mu\text{m}$ separated by sieving. The conversion was measured by complete decarbonation of a sample (2 h in a 950°C furnace).

3. EXPERIMENTAL RESULTS.

The influence of : (i) the electric parameters (U,I), (ii) the gas flow rate (\dot{Q}_g), (iii) and the particle diameter on the rates R_s and X was studied. The variation ranges are the following :

$$150 \text{ A } \leq I \leq 300 \text{ A} \quad (45 \text{ V } \leq U \leq 65 \text{ V})$$

$$10 \leq \dot{Q}_g \leq 20 \text{ l.mn}^{-1} (\text{N}_2)$$

$$d_p = 111 \text{ } \mu\text{m} \text{ and } 172 \text{ } \mu\text{m}.$$

The experimental conditions for the tests are listed on Table 1

dp = 111 μ m								
ESSAI N	X 3	X 4	X 5	X 6	X 7	X 8	X 9	X 10
U (V)	45	50	50	55	50	60	56	65
I (A)	150	150	180	220	150	250	150	300
\dot{Q}_g (l/mn)	10	15	10	10	10	10	20	10
Pu (Kw)	3.25	4.	5	6.6	3.75	8.25	4.65	11.75
Σ_s (Kw h/m ³)	5.41	4.44	8.33	11.	6.25	13.75	3.875	19.58

dp = 172 μ m								
ESSAI N	DC 2	DC 3	DC 4	DC 5	DC 6	DC 8	DC 9	DC 10
U (V)	40	45	50	45	50	65	60	65
I (A)	150	150	180	150	220	250	150	300
\dot{Q}_g (l/mn)	10	15	10	10	10	10	20	10
Pu (Kw)	2.	3.	4.75	3.3	5.1	9.5	5.5	11.88
Σ_s (Kw h/m ³)	3.33	3.33	7.91	5.5	8.5	15.83	4.56	19.8

Table 1 - The experimental conditions.

A typical variation of the bed temperature as a function of time is schemed on figure 3. With fine particles no stable spouted bed is formed, an unstable expanded bed is observed in the whole range of operating conditions. Consequently, the temperature varies with the location inside the reactor and with time, the curve shown on figure 3 must be taken as an indication of the mean variation during the calcination. Four steps are observed : (1) preheating with argon plasma, (2) rapid temperature increase with N_2 plasma, (3) reaction plateau, (4) fast temperature rise before complete reaction due to the decrease of the reaction rate.

Influence of the specific enthalpy and particle diameter (d_p).
 The influence of E_s on conversion and attrition rate is illustrated on figures 4, 5 and 6. Figure 4, a conversion versus time plot, shows the sigmoïde shape of the curves. Complete calcination of $CaCO_3$ is not observed because of the experimental procedure : the test are stopped when a significant temperature increase is measured after the reaction plateau (see figure 3).

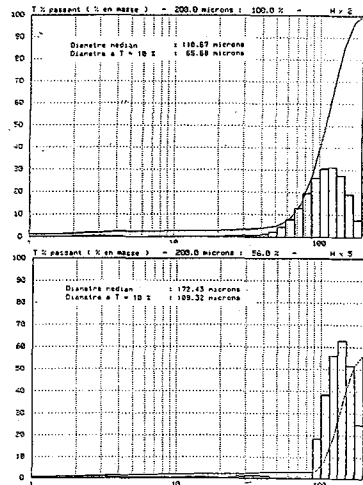


Fig. 2 - Particle size distribution of the calcite.

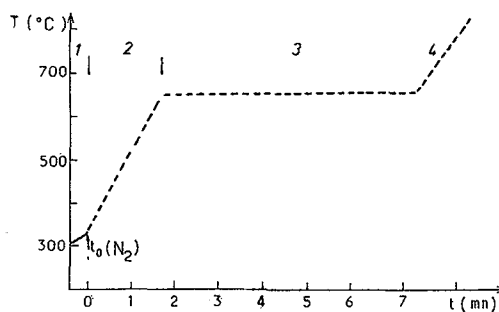
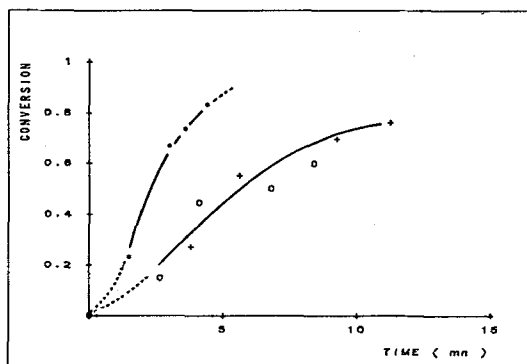
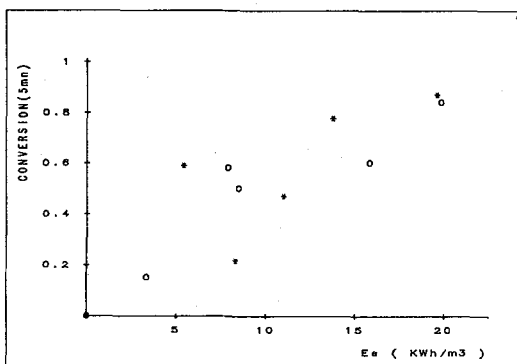


Fig. 3 - Typical temperature variation during a test.



**Fig. 4 - Calcite conversion versus time $d_p = 111 \mu m$,
 $Q_g = 10 \text{ l.mn}^{-1}$
 + : $E_s = 5.4 \text{ kWh.m}^{-3}$,
 O : $E_s = 11 \text{ kWh.m}^{-3}$,
 * : $E_s = 19,6 \text{ kWh.m}^{-3}$.**



**Fig. 5 - Conversion versus enthalpy
 $Q_g = 10 \text{ l.mn}^{-1}$
 * : $d_p = 111 \mu m$,
 O : $d_p = 172 \mu m$.**

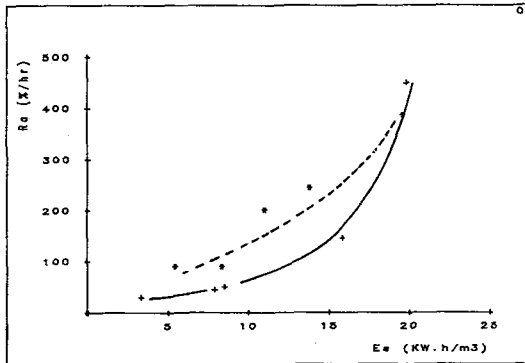


Fig. 6 - Attrition rate versus enthalpy. * : $d_p = 111 \mu m$,
+ : $d_p = 172 \mu m$.

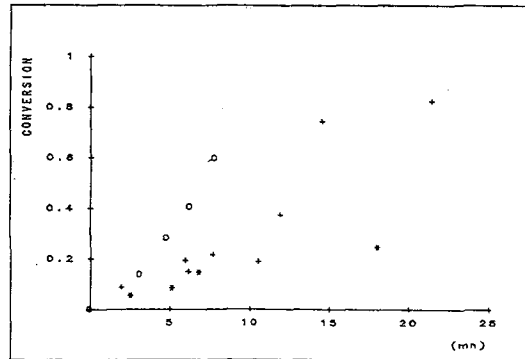


Fig. 7 - Conversion versus time N_2 flow rate as parameter, + 10 l.mn^{-1} , * 15 l.mn^{-1} , O : 20 l.mn^{-1} .

The variation of the conversion at $t = 5$ minutes is illustrated on figure 5. The general trend is an increase of X with E_s but some measurements are affected by the hydrodynamical instability of the bed. No significant differences are observed when comparing the two particle diameters. The effect of specific enthalpy on attrition rate is strong as shown on figure 6. The rate ranges between 30 \% h^{-1} and 100 \% h^{-1} for enthalpies less or equal to 10 kWh.m^{-3} , then large increases are measured : R_a reaches 400 \% h^{-1} for $E_s = 20 \text{ kWh.m}^{-3}$. Attrition rate of small particles ($111 \mu m$) seems to be larger than for $d_p = 172 \mu m$.

Influence of the gas flow rate. The effect of gas flow rate on conversion is plotted on figure 7. Significant variation are measured, for example X increases from 0.2 to 0.5 when Q_g is twice. On the other hand no effect of the gas flow rate on attrition rate is obtained.

Particle size distribution. The variation of size distribution of particles on the reactor as a function of time is shown on figure 8. The increase of the fine particles due to attrition is clearly demonstrated (remember that initial calcite does not contain any particle of diameter smaller than $80 \mu m$). The diameter of fines ranges between 20 and $30 \mu m$, at time $t = 10 \text{ mn}$ 6 % of the particles are smaller than $10 \mu m$ and 6 % larger than $40 \mu m$. The size distribution, at the end of the run, obtained for extreme values of the specific enthalpy is illustrated on figure 9.

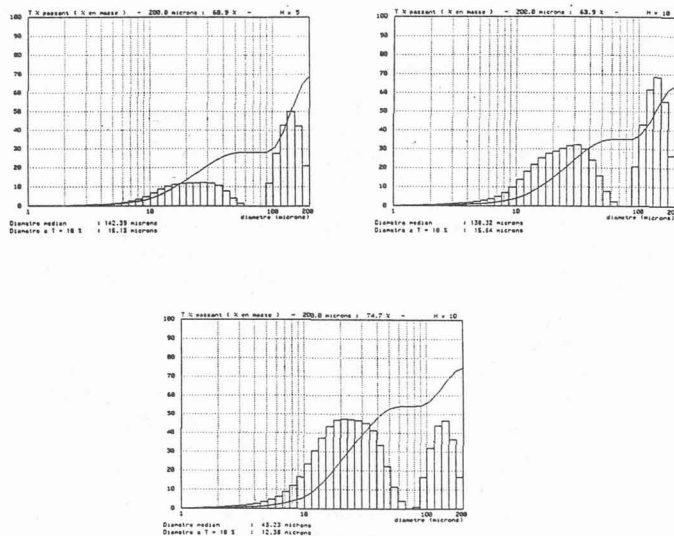


Fig. 8 - Particle size distribution as a function of time.
 $d_p = 172 \mu\text{m}$, $Q_g = 10 \text{ l.mn}^{-1}$, a : t = 1.7 mn, b : t = 4.4 mn,
 C : t = 10 mn.

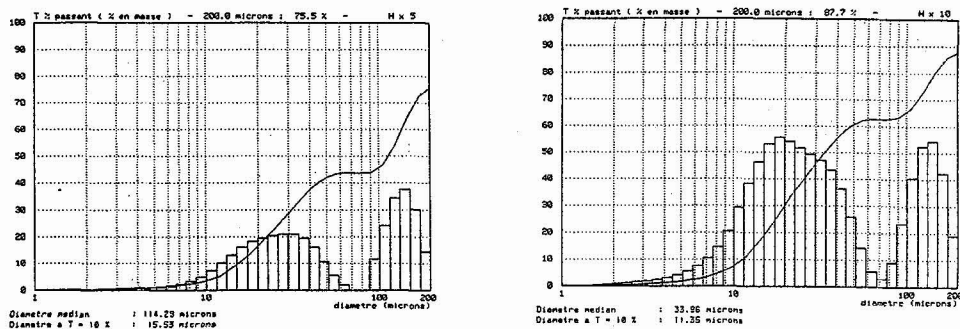


Fig. 9 - Particle size distribution as a function of specific enthalpy
 $d_p = 172 \mu\text{m}$, a : $E_s = 3.3 \text{ kWh.m}^{-3}$, t = 20 mn,
 b : $E_s = 20 \text{ kWh.m}^{-3}$, t = 5 mn.

Both quantity and size are affected by E_s , when E_s increases from 3.3 to 20 kWh.m^{-3} the mean diameter decreases from 30 μm to 20 μm .

No significant variation of fine size distribution is obtained as a function of particle diameter ; in addition the particles collected in the cyclone are size - like the reactor fine particles.

4. DISCUSSION AND CONCLUSION.

A detail examination of the variation of particle size distribution with time gives an indication of the evolution trend of attrition rate during the experiments. The data plot on figure 6 are mean values of R_a at the end of thermal treatment, particle size variation versus time indicates a significant decrease of attrition rate with time. Nevertheless, both measurement techniques are not directly comparable, the first (Fig. 6) is based on weighting, whereas the second is based on the evaluation of the number of particles of size d_p .

The comparison of internal pores size distribution obtained with low and high value of the specific enthalpy /5/ indicates a large increase of the total volume of pores (V_p) and of the pores surface area per unit weight (S_p), in addition the amount of small sizes pores increases, typical values are the following ($d_p = 172 \mu\text{m}$):

$$E_s : 3.3 \text{ kWh.m}^{-3} \rightarrow V_p = 293 \text{ mm}^3.\text{g}^{-1}, S_p = 0.58 \text{ m}^2.\text{g}^{-1}$$

$$E_s : 19.8 \text{ kWh.m}^{-3} \rightarrow V_p = 581 \text{ mm}^3.\text{g}^{-1}, S_p = 1.93 \text{ m}^2.\text{g}^{-1}$$

As a conclusion, the experimental data proves that calcination and attrition occur simultaneously with significant rates during thermal treatment of calcite in a plasma spouted bed reactor. The dominant parameter appears to be the enthalpy of the plasma. For 20 kWh.m^{-3} nitrogen plasma calcination rate of about 1 mole.mn^{-1} and attrition rate of 10 % mn^{-1} are measured. The mean diameter of fines ranges between 20 and 30 μm .

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