



Limestone fragmentation and attrition during fluidized bed oxyfiring

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ABSTRACT

Attrition/fragmentation of limestone under simulated fluidized bed oxyfiring conditions was investigated by means of an experimental protocol that had been previously developed for characterization of attrition/fragmentation of sorbents in air-blown atmospheric fluidized bed combustors. The protocol was based on the use of different and mutually complementary techniques. The extent and pattern of attrition by surface wear in the dense phase of a fluidized bed were assessed in experiments carried out with a bench scale fluidized bed combustor under simulated oxyfiring conditions. Sorbent samples generated during simulated oxyfiring tests were further characterized from the standpoint of fragmentation upon high velocity impact by means of a purposely designed particle impactor. Results showed that under calcination-hindered conditions attrition and fragmentation patterns are much different from those occurring under air-blown atmospheric combustion conditions. Noteworthy, attrition/fragmentation enhanced particle sulfation by continuously regenerating the exposed particle surface.

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1. Introduction

Attrition and fragmentation of limestone during the fluidized bed (FB) combustion of sulfur-bearing fuels have been thoroughly characterized over the last decade [1–7]. Key phenomenological features and mechanistic pathways of sorbent attrition/fragmentation in air-blown atmospheric FB combustors have been disclosed with the aid of a comprehensive test protocol consisting of different and mutually complementary test procedures [1,3,7]. Primary fragmentation occurs as a consequence of thermal stresses and internal overpressures due to carbon dioxide emission. Attrition by abrasion and secondary fragmentation are related to mechanical stresses due to rubbing and collisions with other particles or with the reactor walls or internals. Particle breakage upon impact provides another pathway to sorbent fragmentation, particularly in the jetting region of the bottom bed and in the cyclone inlet ducts. The progress of chemical reactions (calcination, sulfation) interferes with the attrition/fragmentation processes, making the phenomenology even more complicated.

The mechanisms and extent of attrition/fragmentation of sorbents under oxyfiring conditions have only recently received consideration. Under these conditions, which are gaining much interest in connection to carbon capture and sequestration (CCS) techniques, carbon dioxide partial pressures established in the reaction zone may be large to the point of preventing limestone calcination. It has been shown [8] that primary fragmentation is less pronounced under calcination-hindered conditions. Sulfur

capture is also significantly affected, the main pathway being direct sulfation of the raw limestone. Patterns and extent of sorbent attrition and impact fragmentation are also likely to be affected.

Shimizu et al. [5] reported that under pressurized FB combustion conditions (where large CO₂ partial pressures establish) sulfur capture was controlled by the limestone attrition rate. In fact, since most of the limestone in the boiler captures sulfur dioxide at a very slow rate under product layer SO₂ diffusion control, attrition of the sorbent particles surface may reduce the thickness of the calcium sulfate layer and, in turn, increase the reaction rate.

The present study addresses attrition and fragmentation of limestone under simulated FB oxyfiring conditions. Experiments were carried out with a bench scale FB combustor to investigate the extent and pattern of attrition by surface wear in the dense phase of a fluidized bed and the influence of parallel limestone sulfation on this phenomenon. Sorbent samples generated during simulated oxyfiring tests have been further characterized as regards their fragmentation behavior under high velocity impact conditions in a purposely designed particle impactor. In a forthcoming paper [9], the phenomenology of attrition/fragmentation observed under oxyfiring conditions will be compared with that recorded during air-blown atmospheric FB combustion.

2. Experimental

2.1. Apparatus

Sulfation of limestone was carried out in a stainless steel atmospheric bubbling FB reactor. The reactor, 40 mm ID and 1 m high,

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was electrically heated. The gas distributor was a perforated plate with 55 holes of 0.5 mm diameter in a triangular pitch. CO₂, O₂ and a SO₂-N₂ mixture were separately supplied to the reactor by means of digital mass flow meters/controllers. Flue gases were continuously sampled at the exit for CO₂ and SO₂ concentration measurement by means of two on-line NDIR analyzers, in order to monitor the progress of reactions. A two-exit head was purposely designed to convey flue gases through either of two 25 mm ID cylindrical sintered brass filters (filtration efficiency = 1 for >10 μm-particles). Alternated use of filters enabled time-resolved capture of elutriated fines at the exhaust. The bed material consisted of mixtures of 20 g of limestone and either 150 g of silica sand or 200 g of corundum (corresponding to an unexpanded bed height of 8.3 cm). Silica sand was sieved in the two particle size ranges 0.2–0.3 mm and 0.85–1.0 mm, corundum in the particle size range 0.2–0.3 mm. Minimum fluidizing velocity at 850 °C was 0.02 and 0.28 m s⁻¹ for the two sand size ranges, respectively, and 0.03 m s⁻¹ for corundum. Further details on the apparatus can be found elsewhere [1].

Impact testing of pre-processed sorbent samples was carried out in the test apparatus represented in Fig. 1. Particles are entrained in a gas stream at controlled velocity and impacted against a target [7]. The test rig consists of a vertical stainless steel eductor tube (1 m high, 10 mm ID) equipped with a particle feeder. The particle feeding device is fitted at the top of the eductor tube and consists of a stainless steel hopper with a 10 mm ID at the top section and 4 mm ID at the bottom section. The bottom end of the hopper is connected through a valve to a steel tube (6 mm OD and 4 mm ID) running coaxially for 0.2 m inside the eductor tube. Air enters the top section of the eductor tube and flows downwards between the inner and outer tubes. When the valve is opened, limestone particles contained in the hopper flow through the inner tube driven by gravity and by the draw induced by air flowing in the eductor tube. The hopper can be isolated from the environment by means of a top valve, to avoid air bypass when

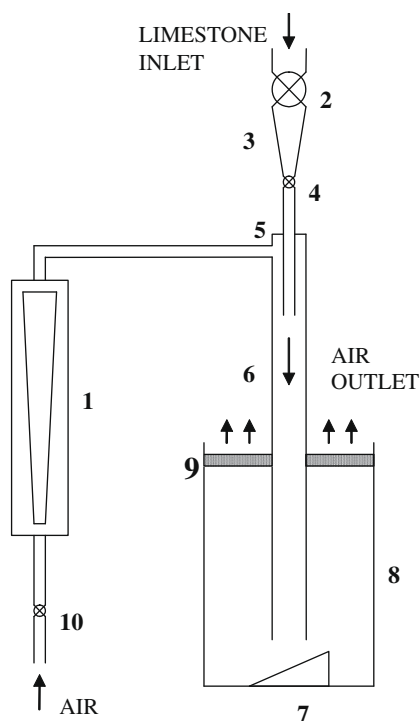


Fig. 1. Impact damage test apparatus. (1) gas flowmeter; (2–4) lock hopper valves; (3) hopper; (5) feeding tube; (6) eductor tube; (7) target plate; (8) collection chamber; (9) cellulose filter and (10) gas flow metering valve.

the bottom valve is open. After feeding, the particles are accelerated by the air flow in the eductor tube. The particle velocity is controlled by regulating the air flow in the eductor tube, by means of a flowmeter. When the particles exit the eductor tube, they impact on a rigid target plate placed in a collection chamber 50 mm below the tube exit. The target is made of stainless steel and is inclined by 30° with respect to the horizontal. This inclination was chosen as a trade off between the need of avoiding interference between the impacting and reflected particles and the need of minimizing the departure of results from those obtained with a target perpendicular to the particle trajectory [10]. The collection chamber is made of a glass vessel, 0.55 m high and 80 mm ID. The air flow leaves the collection chamber from the top section where it passes through a porous cellulose filter (for the capture of finer particulate). The impacted limestone particles settle at the bottom. The device is designed so as to minimize the loss of limestone entrained by air in the chamber, enabling easy collection of the limestone particles after impact for further analysis. The particle impact velocity was calculated as the sum of the gas velocity in the eductor tube and the particle terminal velocity. Particle acceleration to this velocity is complete before impact, as confirmed by particle tracking at the exit of the eductor tube with a high-speed (10,000 frames per second) video camera Photron Ultima APX.

2.2. Procedures

A high-calcium (96.8%) Italian limestone (Massicci) was used in the experiments. Fresh limestone particles were sieved in the particle size range 0.4–0.6 mm, falling well within the range of particle sizes that are customarily employed in practical circulating FB combustion. Limestone particles were then sulfated (at 2000 ppmv SO₂) to completion in the FB reactor operated batchwise at 850 °C with a gas superficial velocity of either 0.51 or 0.73 m s⁻¹. In all the tests CO₂ inlet concentration was kept at 71% by volume. In this environment, at atmospheric conditions, limestone calcination is prevented as verified by XRD analysis of the exhausted sorbent, which did not show any presence of CaO.

At the end of each test the sorbent was first cooled down in a high-CO₂ environment and then discharged from the bed. The sorbent was easily sieved out of the bed material because of its smaller or larger particle size. Sulfated limestone particles were again sieved in the particle size range 0.4–0.6 mm and stored in a desiccator to prevent hydration of the material.

Samples (approximately 2.0 g) of sulfated limestone were weighed and used for fragmentation tests in the impact testing apparatus. The tests were carried out in air at ambient temperature with the following particle impact velocities *v*: 10, 17, 24, 31, 38 and 45 m s⁻¹. These velocities were selected so as to reproduce impact conditions that are likely to establish near the gas distributor or in the cyclone inlet ducts of industrial-scale circulating FB combustors. After each test the sample was retrieved from the collection chamber and weighed. Closure of the mass balance was checked to estimate the loss of material during testing. The closure was always within 3% of the initial sample weight. The collected particles were then sieve-analyzed to obtain their particle size distribution. Selected tests were repeated to check the reproducibility of the particle size distributions.

3. Results

3.1. Calcium conversion degree

Fig. 2 reports the overall degree of calcium conversion X_{Ca} as a function of time during sulfation tests carried out in beds of either

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