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Methodology of mechanical characterization of coated spherical materials

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ABSTRACT

The aim of this work was to develop a methodology for the mechanical characterization of catalyst beads with a core-shell structure and more especially for coated spherical granules. Supports composed of an alpha alumina core coated by gamma alumina shell were shaped by pan coating to this purpose. The proposed methodology started with the characterization of the microstructure of the coating and the highlight of potential macro defects within. Thereafter three tests simulating mechanical stress, such as impact, compression (bulk crushing test), and shear (drum attrition test) are used. The operational parameters of these tests were also optimised in order to stress preferentially the shell of the coated materials. Among the evaluated tests, drum attrition seems to be most efficient for characterizing coated spherical granules.

1. Introduction

Attrition of granular materials is a wide spread phenomena during their life cycle within a large panel of industrial processes. Most of the time, attrition is determined specifically for a type of stress representative of a process step, storage conditions, transport, or handling. Mechanical characterization of the materials allowing to evaluate their strength towards these miscellaneous stresses is then critical. Associated tests should fit the following criteria: simulation of stresses during life cycle of catalyst (shear, impact, and compression), and ability to characterize a significant number of particles of millimeter scale. Drum attrition test, impact test, and bulk crushing test (BCS) are the usual tests being used at IFP that meet criteria for mechanical characterization of spherical or cylindrical catalyst beads.

Last advances in conception of catalyst beads have seen the emergence of core-shell catalysts possessing two catalytic functions [1]. These supports are obtained most of the time by coating a spherical oxide core by another oxide presenting other textural properties. If the tests quoted above are well suited for characterization of classical beads, we wish to check their relevancy for mechanical characterization of coated materials. Furthermore, it should be useful to have a methodology of mechanical characterization in order to study in a systematic way the influence of the coating formulation on attrition strength. The following study is about the optimisation of the mechanical characterization tests for coated materials composed of an alpha alumina core coated with a gamma alumina shell. After achievement of the coating thanks to the use of a granulator pan, the mechanical behaviour of the coated materials has been studied and compared to the initial non-coated core.

2. Experimental section

2.1. Raw materials

Alpha alumina beads (Sphéralite 512 - Axens), sieved between 2.5 and 2.8 mm, were used as a core. The major constituent of the coating is a gamma alumina powder (Puralox - Sasol) with a mass median diameter of 40 µm (dv_{10}=10 µm, dv_{90}=90 µm) and a specific surface area of 210 $m^2\ g^{-1}$. The binder used is a boehmite sol (Pural SB3 - Sasol). The device for coating is a pan granulator GRELBEX P30 equipped with a cylindrical conical bowl. First, 100 g of beads that corresponds approximatively to the use of 8000 beads, are placed in cascade state of flow at rotary speed of 40 rpm and 30° angle. Once the porous volume of Sphéralite 512 is filled with the boehmite sol by pulverisation, 10 g of gamma alumina powder is continuously added under sol pulverisation. After adding the precursors, the coated core are dried in a ventilated drying oven at 30 °C during two days, then calcined in a muffle furnace at 600 °C for 2 h in air with a heating rate of 3 °C/min.

2.2. Characterization

The first step of our methodology is to establish the structure of coated materials (textural properties, microstructure, presence of macro defects). All of the characterizations are performed on calcinated materials. The textural properties are determined by nitrogen physisorption (ASAP 2420 - Micromeretics) and mercury porosimetry (Autopore 4 - Micromeretics). The specific surface area is obtained by B.E.T mathematical treatment. The microstructure of coated materials and particles generated after mechanical tests are

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Fig. 1. Steps for image analysis a) initial image, b) binarised image (cracks are in white), c) detection of cracks (each crack is in different colour). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

analyzed by SEM (6300 and 6340F-JEOL). The occurrence of macro-defects inside the coating are also important for understanding the mechanical behaviour of a material. A software for image analysis called LoCaFi has been coded for this purpose (automatic determination of number, area, and circularity (ϕ=4×π× area/perimeter) of cracks). The method of defect detection uses an image binarisation (Fig. 1), an assembly of connected zones, then an accounting of cracks with determination of their area and their circularity. This treatment is performed about on average of sixty micrographs of coating in order to get statistically significant data.

Then, it is wished to develop a mechanical characterization procedure for coated spherical material. Generally, catalyst beads received a pre-sieving to eliminate fine particles on their surface before mechanical characterization. It has been observed that sieving constitutes already a mechanical test as the latter leads, according to its intensity and duration, to an important generation of fine particles for our materials. Therefore this step has been withdrawn for a better qualification of mechanical tests' contribution to attrition. Also, the follow-up of attrition rate during the tests needs at least a coarse separation of fines particles and initial beads. This is done on a sieve shacked twice manually. At the end of each test, the coated material is sieved with a weak intensity during 1 min on a sieving machine Retsch AS-200. The oversize and undersize particles will be called respectively “mother particles” and “cuttings”. The studied measurement parameters are the attrition rate ξ and the normalized attrition rate *E*, defined as follow:

$$\xi = 1 - \frac{M_a}{M_i} \quad (1)$$

$$E = \frac{\xi_e}{\xi_s}. \quad (2)$$

With M_a the mass of mother particles, M_i the initial mass of coated material, ξ_e and ξ_s the attrition rate of coated materials and cores respectively. The normalized attrition rate will be useful to compare relatively the different tests and find the best operational parameters. Indeed an increase of value of *E* means an increase of the component of the stress applied to the coating compared to the component applied to the core.

The impact test has the advantage of characterizing a great number of particles with strain velocity similar to those met into industrials unit during loading of reactors, unloading of silos, pneumatic transport, and this, in a lapse of time relatively short compared to the standard method using a compression test. The impact tests are performed on a device elaborated by university of Surrey comparable to that used by Yuregir [2] (Fig. 2). Particles are introduced individually thanks to a vibratory distributor inside a tube. The particles are accelerated by a flow of air to undergo an impact against a target constituted of Duplex steel. The angle of the target can be oriented between 0° and 60°. The velocity of particles is determined just before the impact by measuring the time of flight between two photodiodes. Cuttings and mother particles are collected inside a chamber maintained under vacuum. The intensity of the vacuum inside the collection chamber allows to control the flow of air needed to accelerate the particles. Each test is performed using a single impact with a given velocity and angle. A mass of 30 g corresponding to the use of 2500 coated beads used for each test. The impact angle of 0°, 30°, 45° and 60° have been studied, and for each angle, the coated materials have undergone an impact velocity of 3, 5, 8, 11, 14, and 21 m s^{-1}. The attrition rate is evaluated at the end of each test with a 2 mm sieve.

The drum attrition test simulates the shear stress inside silos, moving bed, and flourishing bed. The standard ASTM [3] recommends

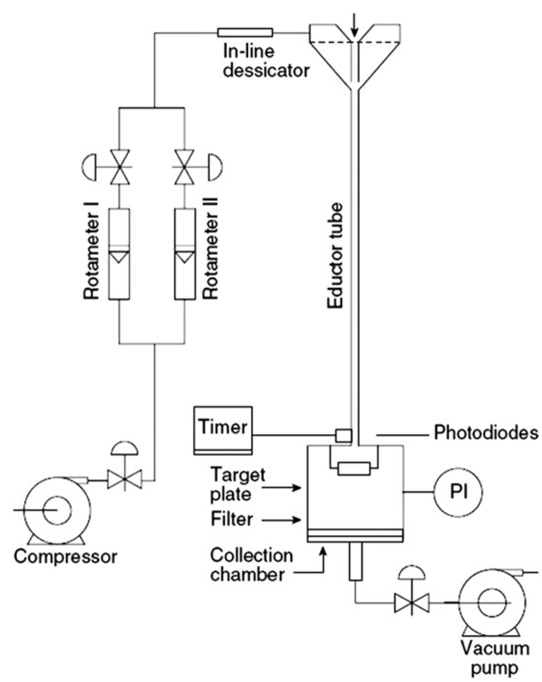

Fig. 2. Diagram of the impact test.

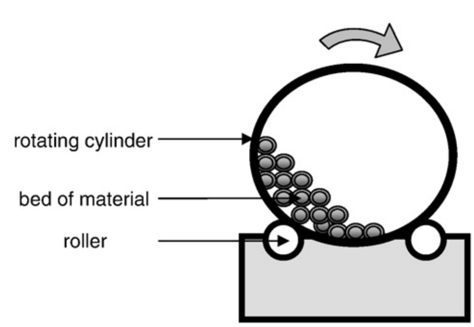

Fig. 3. Diagram of the drum attrition test.