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Hydrodynamic Parameters Study of Gas-Solid Fluidized Bed Reactor: Case of AUC Conversion to UO2

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ABSTRACT

The fluidization technique is used for gas-solid interaction processes whenever high rates of heat and mass transfer between the two constituents is required. This work aims to contribute to the understanding of the hydrodynamic parameters of gas-solid fluidized bed reactors in the case of the conversion of AUC powder (ammonium uranyl carbonate) into UO_2 powder (uranium dioxide). The study focuses on the fluidization velocity effect on the pressure drop evolution during the fluidized bed process, as function of the temperature and type of gas used (pure N_2, pure H_2, and a 50% H_2–50% N_2 mixture). The study will also present the results of the gas flow rate variation as a function of temperature, as well as the results of the pressure drop variation as a function of the fluidization velocity, taking temperature as a parameter for pure N2, pure H2 and 50% H2-50% N2 mixture. Calcination-reduction experiments were conducted, using a fluidized bed reactor to convert AUC powder into UO2. These experiments examined the influence of process parameters on particle size, specific surface area, O/U ratio and porosity of the UO2 powder produced. Using the optimized operating parameters, such as the fluidization velocity, gas flow rates, and treatment temperature of the fluidized bed reduction-calcination process, ensured the production of UO_2 powders with the required physicochemical characteristics.

Keywords: fluidized bed reactor; fluidization velocity; pressure drop; calcination; reduction; AUC; UO_2.

Graphical abstract
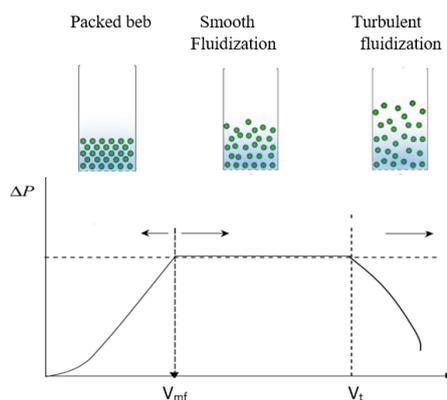
1.   Introduction

Fluidization refers to the process by which particles are suspended due to the action of a fluid passing through them, such that the entire set of particles also tends to behave like a fluid [1-6]. From a macroscopic perspective, the solid phase (or dispersed phase) behaves like a fluid, hence the origin of the term "fluidization". The collection of fluidized particles is known as a fluidized bed.

Fluidization is a technique widely used worldwide in laboratories and industrial plants, in the most varied processes. One of the advantages brought by fluidized bed systems in industry is temperature homogeneity. With a fluidized bed system, it is possible to perform a wide variety of heat treatments, simply by changing the composition of the gas mixture. Fluidized bed systems are used in various types of heat treatments, such as the uranium dioxide production from uranium tetrafluoride, uranium Hexafluoride and ammonium uranyl carbonate in fluidized bed furnace [7-10].

When a fluid passes vertically through a bed of particles, the pressure drop across the bed ΔP will increase proportionally to the superficial velocity V of the fluid, until it reaches a value V_{mf} which is the minimum fluidization velocity, above which the pressure will remain constant. Increasing the superficial velocity beyond this value will result in a continuous expansion of the bed until eventually entraining the particles, if they are not physically bound to each other. The fluid velocity at which these conditions are reached is called the minimum fluidization velocity and the bed of particles is known as a fluidized bed. Fluidized bed hydrodynamic behavior is very complex and must be understood to improve fluidized bed operations. One of the most important parameters to characterize fluidized bed conditions is the minimum fluidization velocity (V_{mf}) [11-14].

The hydrodynamic behavior of fluidized systems, has been the subject of numerous studies by various researchers, due to the scientific and practical interest in the expansion of a fluidized system [15-21]. According to Matsen J.M. [22], the expansion of a bed of solid particles fluidized in gas is a function of the surface velocity of the gas, which is perhaps one of the most important properties of the system.

Depending on their characteristics, the fluidized bed can be classified as particulate or aggregate. A bed is said to be particulate if the volumetric concentration of solid particles, that is, the number of particles per unit volume, is uniform throughout the bed and does not vary with time. The system is said to be aggregative if, at a constant rate of the fluidizing medium, the volumetric concentration of solid particles is not uniform and at a given point the concentration varies with time [23, 24].

In a gas-fluidized bed the particles are supported above a gas distributor plate which permits the gas to enter and flow upward through the bed. As the gas flow increases, there will be a pressure drop across the bed which will eventually reach a point when it just balances the downward gravitational force of the particle bed. When this point is reached, the bed will expand and the particles separate. A distribution plate is a crucial component in a fluidized bed reactor that sits beneath the bed of solid particles. Its primary function is to uniformly introduce the fluidizing gas or gas mixture across the bed, preventing particle backflow and ensuring proper contact between the gas and solids. Different designs, such as perforated plates and bubble caps, are used to influence the hydrodynamics, pressure drop, and overall performance of the reactor [1, 2, 25-27].

A distribution plate is a crucial component in a fluidized bed reactor, which is used to process materials like uranium dioxide. This plate, often a perforated grid, is placed at the bottom of the reactor and distributes the upward-flowing gas uniformly to suspend and fluidize the uranium dioxide particles, ensuring a uniform temperature and efficient reaction. This process is vital for reactions involving uranium dioxide, which can be exothermic and require precise temperature control to prevent particle sintering or agglomeration. The use of fluidized media in processes with significant thermal effects is due to the high transfer rate that characterizes these media.

The transfer is so intense that it occurs over a layer only a few particle diameters above the distributor. The intensive mixing of the solid phase particles, caused primarily by bubbling, results in a uniform heat distribution. The transfer between different points in the layer is very rapid because of the large exchange surface area provided by the finely divided solid [28, 29].

In this study, experimental and theoretical methods are used to determine the minimum fluidization velocity and the pressure drop evolution as a function of gas flow rate (N_2 and H_2), fluidization velocity and temperature. The results are discussed in terms of the characterizations performed on the UO_2 powders produced in accordance the experimental protocol adopted for the conversion process of the AUC powder into UO_2 in a fluidized bed furnace, in comparison with the

required specifications of uranium dioxide pellets cited in the literature [28, 30-31].

2.   Materials and Methods

2.1.Fluidized bed furnace equipment

The fluidized bed furnace, consist of a stainless steel tube surrounded by two heating elements with the capacity to reach a temperature of 700 °C. The fluidization experiments were carried out with a distribution plate made of sintered stainless steel powder. At the inlet of the system, a rotameter measures the gas flow rate and a pressure gauge measures the pressure in the pipe. The distribution plate is located at the bottom, next to which is a pressure gauge that measures the pressure under the distribution plate. At the top of the column is a cylindrical filter, also made of sintered stainless steel powder, used to retain particles carried by the gas flow. Another pressure gauge is placed at the top of the column to detect an increase in internal pressure due to possible filter clogging.

Nitrogen (N_2) and Hydrogen (H_2) gases were used as fluidizing gases. For these experiments, the gas flow rate was varied from 0 to 80 L/min and temperature from 20 °C to 650 °C.

The reactor has a thermocouple well inside, so that its internal temperature can be measured. The distributor plate is located in the lower part, which promotes a random flow of gas for fluidization. In the upper part of the reactor, there is a conical chamber that aims to reduce the speed of the fluidizing particles and increase the area for placing instruments and filter elements to retain fine UO_2 particles.

The specific surface area is measured by the BET method using ASAP surface area analyzer (Micromeritics); the average particle size and the distribution of the particle size were measured with laser granulometer analyzer (master size). The O/U ratio measurements by UV–Vis spectrophotometer equipment.

2.2. Methods

Experiments on the calcination-reduction of AUC to UO_2 in Fluidized bed furnace

In order to study the process parameters and their influence on the characteristics of the UO_2 powder, experiments on the calcination of AUC were carried out on the fluidized bed reactor.
From the decomposition of uranyl ammonium carbonate and UO_3 reduction, UO_2 powder was produced. This is necessary, due to the strict specifications of the final product, sintered UO_2 pellets for nuclear fuel.
The process for obtaining UO_2 from AUC by fluidized bed is carried out batch wise and consists of four steps:
   - fluidized bed furnace preparation and introduction of AUC into the furnace ;
   - AUC calcination under a Nitrogen atmosphere ;
   - UO3 and U3O8 reduction under Hydrogen atmosphere ;

In the reduction stage, the fluidized bed furnace is kept under a Hydrogen atmosphere in order to transform the UO_3 and U_3O_8 into UO_2 powder.
The AUC conversion to UO_2 experiments were carried out in accordance the following operating conditions:

The AUC calcination stage was carried out with heating to 400°C under 40 L/min Nitrogen flow rate during two (02) hours. The first reduction stage was carried out at a temperature ranging from 450-570 °C, under a mixture atmosphere with 60 L/min Nitrogen flow rate and 5 L/min Hydrogen flow rate during 30 minutes. This stage corresponds to the reduction of UO_3 to U_3O_8. The second reduction stage was carried out at a temperature ranging from 600-610 °C, with 30 L/min Nitrogen flow rate and 10 L/min of Hydrogen flow rate during 15 minutes. Stabilization stage consists of gradually injecting air with Nitrogen until the furnace cools to approximately 80°C.

2.3. Hydrodynamic Fundamentals of Fluidization

A gas moving upward through a fixed bed of particles experiences a pressure loss due to frictional resistance, which increases with increasing velocity. However, a point is reached where the buoyancy exerted by the gas on the particles

equals their apparent weight in the bed. At this point, the particles are supported by the gas, so the separation between them increases, and the bed begins to fluidize [19-21]. Therefore, the onset of fluidization is associated with a pressure loss of the gas along the bed equal to the apparent weight of all its particles, per unit area of bed perpendicular to the direction of the weight.

2.3.1. Particle Reynolds Number

This parameter is very important because it determines the flow regime. Using equation 1, the Reynolds number of the particles was calculated for fluidization gas velocity values from 0 to 50 cm/s, with Nitrogen atmosphere as the fluidization gas at temperature of 20°C [1-4, 24]:

$$N_{Re} = \frac{\rho.V.d_p}{\mu} \qquad (1)$$

Where: ρ is the gas density (g/cm³), V is the gas velocity (cm/s), d is the particle diameter (cm) and μ is gas viscosity (g/s.cm).

2.3.2. Pressure drop curve as a function of velocity for gas-solid system

The upward movement of gas through a bed of particles causes a pressure drop due to the resistance offered by the particles as the gas passes through. Fig 1 shows the relationship between the pressure drop in the bed and the gas velocity in the gas-solid systems. Starting from the velocity V = 0, it can be seen that as the gas velocity increases, the pressure drop in the bed increases linearly. If the velocity is increased slightly, the pressure drops to just enough to support the weight of the particles. These conditions are called minimum fluidization conditions. Fig1 shows two clearly differentiated sections:
Fixed bed: the pressure drop increases linearly with the surface velocity of the gas. The pressure drop is determined using Ergun's equation (eq. 2) [24]:

$$\frac{\Delta P}{L} = 150 \frac{\mu .V (1-\varepsilon)^2}{d^2 . \varepsilon^3} + 1.75 \frac{\rho V^2 (1-\varepsilon)}{d .\varepsilon^3} \qquad (2)$$

Where: *ρ is fluid density, μ is fluid viscosity, d is particle diameter, L is bed height, Ɛ is bed porosity and V is velocity of the fluid.*

Fluidized bed: once the bed is fluidized, the pressure drop in it remains constant and is equal to the weight per unit area of the particles in suspension in the bed [1-4, 24]:

$$\Delta P = \frac{m .g}{A} \qquad (3)$$

where *: m is the bed particles mass (g), A is across-sectional area (cm²) and g is the gravitational acceleration (9.834 m/s²).*
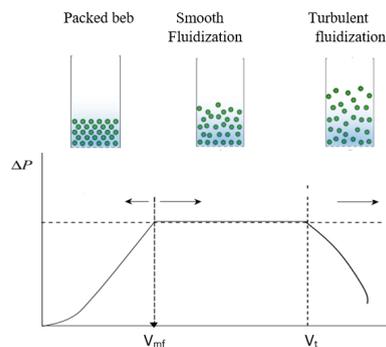

Fig 1. Fluidization types and pressure drop versus velocity curve for gas-solid systems

2.3.3. Minimum fluidization velocity (V_{mf})

Minimum fluidization velocity is one of the most important parameters when characterizing the hydrodynamics of a fluidized bed. Determining V_{mf} essentially involves the flow that produces a pressure drop in the bed equal to its weight per unit section. This is a valid approximation, since it has been experimentally observed that the value of ε (porosity) is between 0.4 and 0.5 in the fluidization of spherical particles of uniform size [1-4, 24].

Assuming a bed of particles with density ρ_p, with a height L in a tank with cross section A, where ε is the void fraction and ρ_g is the density of the fluidizing gas, the pressure drop through it is defined as [1-4, 24]:

$$\Delta P = (\rho_p - \rho_g) . L . (1 - \varepsilon) . g \qquad (4)$$

For small particles, i.e. for low Reynolds number, the minimum fluidization velocity is given by the equation [24]:

$$V_{mf} = d_p{}^2 . (\rho_s - \rho_g) . \frac{g}{1650\,\mu_g} \qquad N_{Re} < 20 \qquad (5)$$

To predict the vapor viscosity of gas mixtures at pressures (N_2+H_2) as a function of temperature, the Bromley and Wilke equation was used, a modification of the Hirschfelder model, described in the Chemical Engineering Handbook Perry [32]. The critical constants of the gases are required. Temperature, pressure, and volume, which were taken from ÀGÀ, Gas Handbook [33].

3.   Results and Discussion

3.1. Particle Reynolds Number determination

This parameter is very important because it indicates the flow regime. Using the values given in Table 1, the Reynolds number of the particle with Nitrogen as the fluidizing gas at temperature values 20°C et 650°C for given gas velocity = 10 cm/s is equal to 0.20 and 0.03, respectively. With these values obtained for the Reynolds number Re < 20, the regime is considered clearly laminar. Using equation 1, the variation in Reynolds number as a function of gas velocity shown in Fig 2 shows that Re values remain below 1 for gas velocities reaching 50 cm/s.

Table 1. Particle Reynolds Number parameters

Properties	Values
UO$_2$ particle diameter (μm)	30
Particle density (g/cm^3)	11
Viscosity N$_2$ 20°C (Cp)	0.017
Viscosity N$_2$ 650°C (Cp)	0.039
Density N$_2$ 20°C (g/L)	1.17
Density N$_2$ 650°C (g/L)	0.36
Fluidization velocity (cm/s)	10
N$_{Re}$ 20 °C	0.20
N$_{Re}$ 650 °C	0.03

3.2. Theoretical calculation of minimum fluidization velocity (Vmf)

Since the regime is laminar, the equation 5 is used for the calculation of the minimum fluidization velocity; where this velocity is a function of the particle diameter, the density of the solid and the viscosity of the gas. Fig 3 shows the variation of V_{mf} as a function of particle diameter. The dependence of the minimum fluidization velocity is strongly affected by particle size. It is noted that V_{mf} increases as a function of particle size.
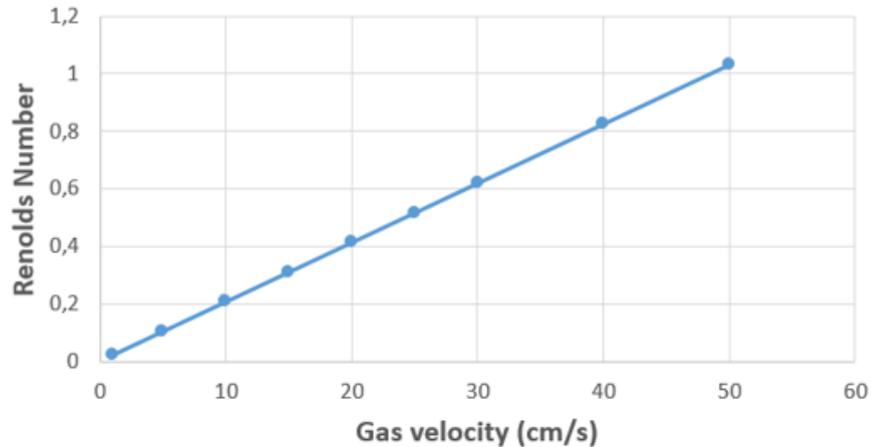
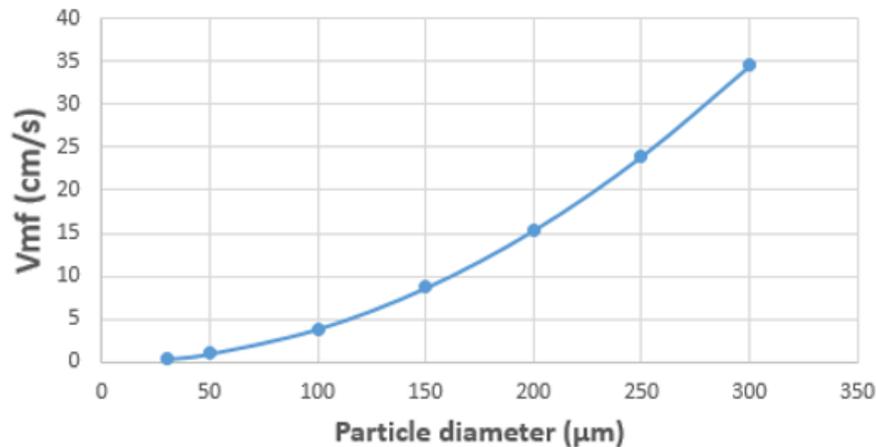Fig 2. Reynolds number as a function of gas velocity (20°C)

Fig 3. V_{mf} as a function of particle diameter

3.3. Effect of the flow rate (gas velocity) on pressure drop

The effects of the flow rate on pressure drop are reported in Fig 4 and 5 for fluidized and fixed beds. Fig 6 and 7 show the graphs of pressure drop versus gas velocity. The effects of flow rate on pressure drop are shown in Figures 4 and 5 for fluidized and fixed beds. Fig 4 corresponds to experiments series carried out on different batches of AUC powder (AUC/1 to AUC/6). Fig 5 corresponds to experiments series carried out on different batches of UO2 powder (UO2/1 to UO2/6).Fig 6 and Fig 7 show graphs of pressure drop as a function of gas velocity.
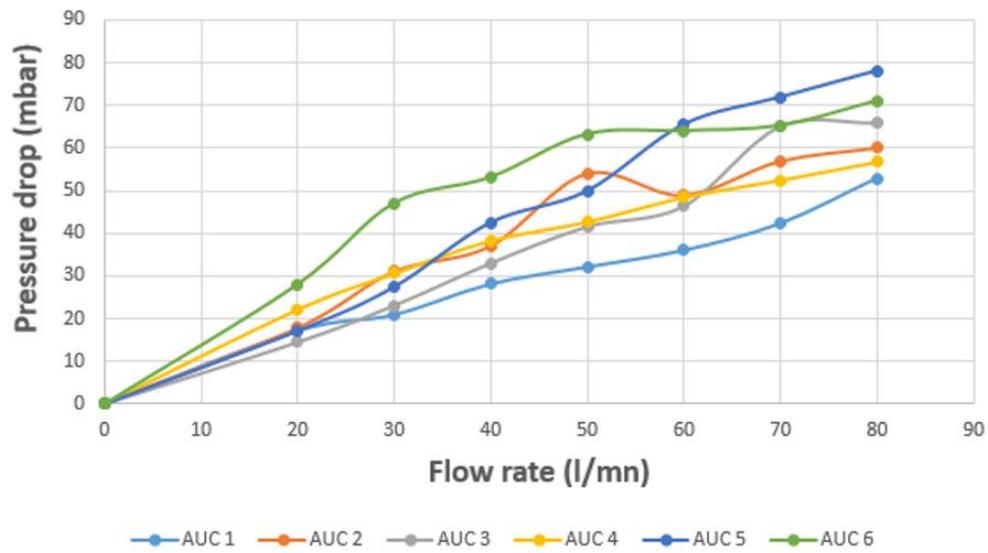
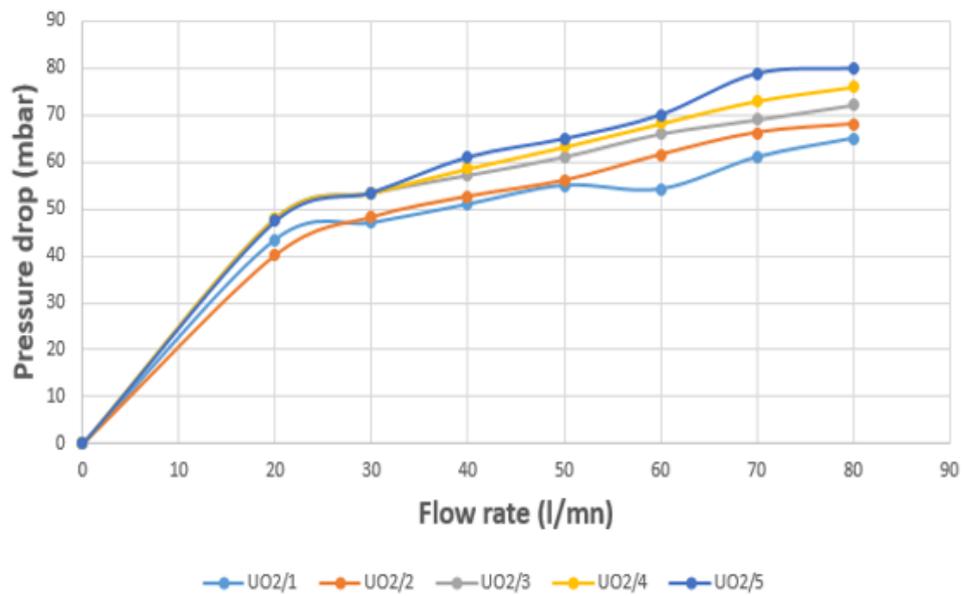Fig 4. Pressure drop variation as a function of flow rate (AUC)

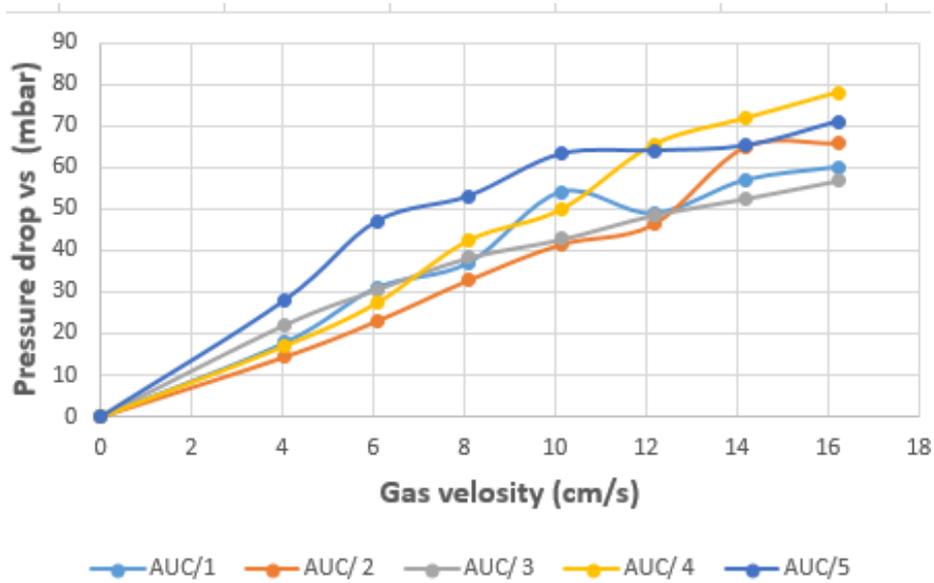Fig 5. Pressure drop variation as a function of flow rate (UO_2)

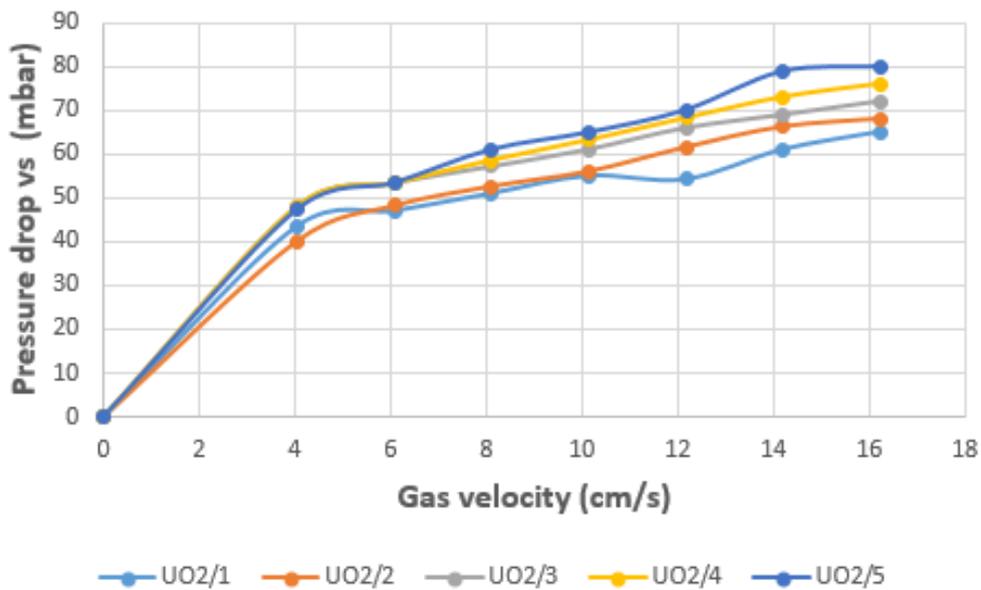Fig 6. Pressure drop variation as a function of gas velocity (AUC)

Fig 7. Pressure drop variation as a function of gas velocity (UO$_2$)

The experimental calculation from the pressure drop curves as a function of the gas velocity, gives a value of the minimum fluidization velocity around 4 cm/s, corresponding to the flow rate value 40 L/mn.

3.3.1 Distribution plate curves: AUC

In the case of the AUC powder, it is observed that a continuous increase in pressure drop across the entire flow range of 0-80 L/min of N$_2$ (0-16.22 cm/sec). The pressure drop deviates from the value corresponding to the static pressure of the bed. This can be attributed to the loss of energy due to collision and friction between particles and the container surface.

3.3.2. Distribution plate curves: UO$_2$

For the UO_2 powder, the pressure drop curves as a function of gas flow rate clearly show a constant pressure drop above the minimum fluidization velocity (4 cm/s) above the minimum fluidization velocity, indicating good fluidization (fluidized bed), starting at a minimum fluidization velocity of around 4 cm/s.

3.4. Effect of temperature on pressure drop

Fig 8 and 9 show the pressure drop variation as a function of temperature, for AUC calcination and UO_3 with U_3O_8 reduction process. It can be observed that the pressure drop is affected by the temperature condition. An increase in pressure drop with increasing temperature is observed for the AUC calcination process under N_2 atmosphere. The pressure drop decreases with increasing bed temperature, in UO_3 process under H_2 atmosphere. As the viscosity of the gases increases with temperature, the differential pressure ΔP of the plate also increases. The variation in the differential pressure ΔP of the plate as a function of fluidization velocity is shown below, taking temperature as a parameter for pure N_2, pure H_2, and a 50 % H_2-50 % N_2 mixture.

The curves for AUC calcination process (Fig 8), show a maximum pressure drop gradient of around 50 mbar. This value indicates reduced fluidization, which may be due to uneven mixing in the particle bed. The curves for UO_3 reduction (Fig 9) show a lower pressure drop gradient, indicating better fluidization quality.
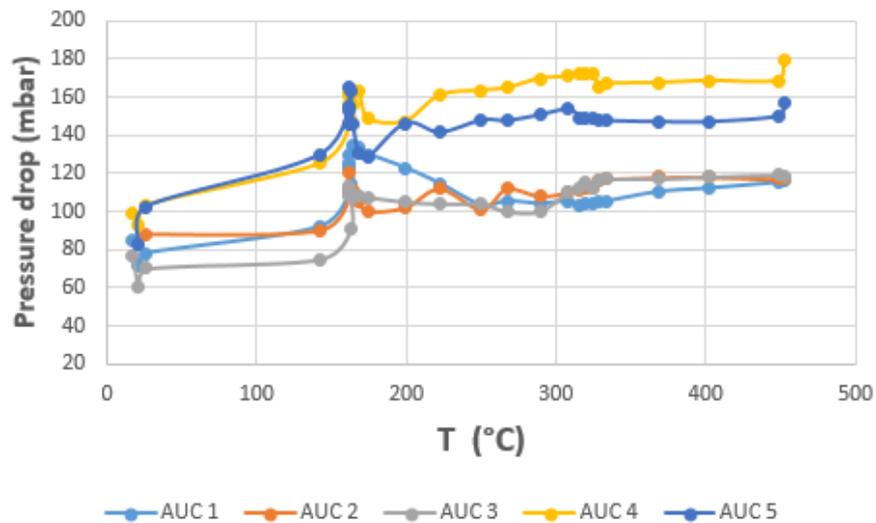

Fig 8. Pressure drop variation as a function of temperature (AUC calcination)
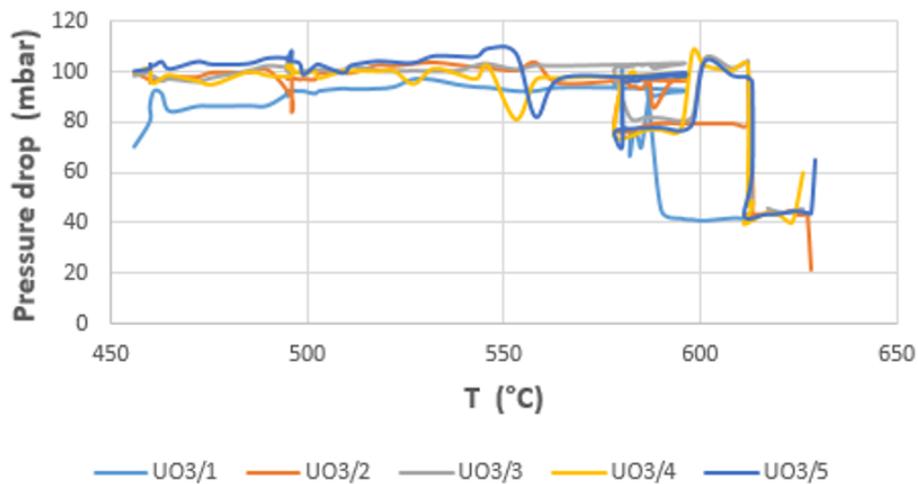

Fig 9. Pressure drop variation as a function of temperature (UO_3 and U_3O_8 reduction)

3.6. Reaction mechanism

3.6.1. Calcination

The calcination of AUC occur under Nitrogen atmosphere with decomposition temperature between 160 and 180 °C, the product obtained: UO_3 (Fig. 8).

$$(NH_4)_4[UO_2(CO_3)_3] \text{ (s)} \longrightarrow UO_3 \text{ (s)} + 2H_2O + 3CO_2 \text{ (g)} + 4NH_3 \text{ (g)} \qquad (6)$$

3.6.2. Reduction

The first reduction of UO_3 to U_3O_8 occur under Hydrogen atmosphere at the temperature between 450 and 570 °C, with gas mixture (N_2+H_2). The second reduction of U_3O_8 to UO_2 under H_2 atmosphere at temperature between 580 and 600 °C.

$$3UO_3 \text{ (s)} + H_2 \text{ (g)} \longrightarrow U_3O_8 \text{ (s)} + H_2O \qquad (7)$$
$$U_3O_8 \text{ (s)} + 2H_2 \text{ (g)} \longrightarrow 3UO_2 \text{ (s)} + 2 H_2O \qquad (8)$$

According to TGA/DTG studies [28-31], have shown that the crystallization of amorphous UO_3 to α-UO_3 occurs in the temperature range of 400–480 °C. UO_3 is reduced to U_3O_8 in the temperature range of 490–600 °C. An exothermic crystallization appears in the 400–480 °C range, and UO_3 is formed. At 490 °C, U_3O_8 begins to form, continuing up to 590 °C. The TGA-DTA curves obtained under a Hydrogen atmosphere, the final product of the reduction of U_3O_8 results from an exothermic reaction. U_3O_8 forms first in the temperature range of 530–570 °C, followed by UO_2 between 550 and 610 °C. The experimental tests demonstrated that the reduction temperature of 580 °C is sufficient for the complete reduction of UO_3 to UO_2. The results demonstrate that the reduction time directly affects the activity of the product.

3.7. Specifications for uranium dioxide powder

UO_2 powders for sintering must have a set of specific physicochemical properties that allow the manufacturing process of the tablets to be defined. This set of properties must target the sinterability of the powder, that is, it must be directly related to the sintering mechanism.

In the sintering process of UO_2 pellets, the main parameters that determine the densification mechanism are: chemical activity (impurities), density and average particle size, porosity, Oxygen/Uranium (O/U) ratio and specific surface area. It should be noted, however, that these parameters are interdependent.

The specific surface area of the UO_2 powder was determined using the Micromeritics device, based on the measurement process developed by Brunauer, Emmet and Teller [34]. A curve is obtained at constant temperature, relating the amount of gas adsorbed to the pressure. This curve is called the adsorption isotherm and through it the specific surface area of the analyzed powder is calculated.

Table 2 shows some characterization results obtained for the produced UO_2 powders following experiments carried out on the calcination-reduction of AUC in a fluidized bed furnace (O/U ratio, specific surface area and particle size distribution).

Influence of process parameters on the properties of UO$_2$ powder

Optimizing equipment operating parameters is of great importance, as pellet quality is strongly influenced by the characteristics of AUC powder, which are themselves correlated with precipitation conditions. AUC is characterized by a uniform particle size and rounded shape.

Both AUC and UO_2 are in granular form, and their particle size distribution is almost identical above 10 μm. This similarity in shape clearly indicates that the AUC particles are converted to UO_2 without breakage during the calcination-reduction stage. Generally, the resulting UO_2 powder has a bimodal size distribution, consisting mainly of fine pores (0.02-0.2 μm) and large pores (02-10 μm) [31, 35-38].

Obtaining UO_2 powder conform to the required specifications is closely linked to the operating conditions for calcining AUC powder and reducing UO_3 and U_3O_8 powders, hence the need to optimize the hydrodynamic and thermal parameters of the fluidized bed process. A desired specific surface area can be obtained by controlling the thermal agitation time and temperature during calcination-reduction, as well as by controlling the internal stresses of the AUC particles during precipitation.

Table 2. Characterization results for the UO_2 produced powders

Properties	Experience 1	Experience 2	Experience 3	Experience 4
O/U ratio	2.09	2.12	2.11	2.08
Specific surface area (m^2/g)	6.82	7.8	8.2	9.02
Particle size distribution (µm)	41.75	45.02	43.14	38.89

4. Conclusion

Based on the results discussed in this work, the following conclusions can be drawn:
- The experimental tests conducted successfully determined the terminal velocities for different flow rates of the continuous phase and established their relationship with the pressure drop within the system.
- The fluidization curve obtained from this study is consistent with those reported in the literature, demonstrating the correlation between the flow rate and velocity of the continuous phase and the pressure drop in the system.
- The fractions of the continuous phase progressively increase within the fluidized bed system as the flow rates of this phase rise, while an opposite trend is observed in the behavior of the dispersed phase.
- The minimum fluidization velocity and the terminal velocity are key phenomenological characteristics of the fluidized bed in multiphase flow systems, and their calculations are essential for the design and development of fluidized bed projects.
- The classification of fluidization provides a framework for defining the type of fluidization and analyzing the behavior of fluids in multiphase flow systems, contributing to the understanding and optimization of such processes.
- The experimental work carried out in this study to convert AUC into UO_2, based on a literature review of gas-solid fluidization systems and heat treatment studies, indicates that the UO_2 powders produced are conform to the desired specifications for uranium dioxide pellets production, in terms of specific surface area, particle size and stoichiometry.

Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflict of Interest

The authors declare that they have no conflict of interest.

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