A new technique for characterizing aerated flocs in a flocculation–microbubble flotation system

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A B S T R A C T

A technique named Aerated Flocs Characterization (AFC) was developed and validated to characterize multiphase systems that are formed in flocculation–flotation (with microbubbles) systems at laboratory scale. Synthetic polyacrylamides were used to flocculate dispersed particles models of kaolin, activated carbon and iron hydroxide colloidal precipitate, Fe(OH)₃. Aggregation and solid/liquid separations were conducted in a dissolved air flotation (DAF) apparatus, which consisted of a flotation cell connected by a release needle valve to a pressure vessel, employed for water saturation at 4 atm and microbubbles formation. The aerated flocs exhibited very rapid rising rates (>60 m h⁻¹) as a result of bubbles adhesion, bubble surface nucleation and bubbles growing and entrapped inside the flocs. All these conformations may adhere or lead to the formation of very light (low density) flocs. The number of bubbles attached or entrapped inside the flocs determines the aeration degree in the so-called aeroflocs, property which depends, among others, on solids type (hydrophobicity); this being very noticeable when compared to activated carbon particles with the less hydrophobic model suspensions. The size distribution, up-rising rates, shape factor, fractal dimension and density of the flocs were determined using this image analysis technique. In addition, the bubble positioning, floc structure and bubble size were monitored. The AFC technique was found to be very reliable showing high statistical reproducibility and appears to have a good potential to characterize particles, aerated flocs and bubbles.

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1. Introduction

Flocculation followed by flotation with microbubbles (Dissolved Air Flotation-DAF) has been widely applied industrially, mainly in water and wastewater treatment contexts, with some advantages over settling or filtration (Bolto et al., 1996; Rubio et al., 2002; Metcalf and Eddy, 2003; Rubio, 2003; Dobiás and Stechemesser, 2005; Bolto and Gregory, 2007; Rubio et al., 2007; Bratby, 2008).

In flotation with microbubbles, a number of bubble/particle (aggregates) interactions have been proposed, namely adhesion of bubbles, bubbles nucleation, entrapment and entrainment (Solari and Gochin, 1992; Rubio et al., 2002; Rubio, 2003; Carissimi and Rubio, 2005a; Rodrigues and Rubio, 2007).

The formation of aerated flocs (structures with high rising rates) has been observed by some authors who describe these flocs as having a large size, low density, rapid up-rising rates, low moisture content and high resistance to shear (Owen et al., 1999; Rulyov, 1999; Colic et al., 2001; Miller, 2001; Rulyov, 2001; Rubio, 2003; Carissimi and Rubio, 2005a; Da Rosa and Rubio, 2005; Rodrigues and Rubio, 2007; Rubio et al., 2007). Such features allowed a very rapid separation of flocs from water allowing high throughput in wastewater treatment processes. Many applications have been published and optimized for futuristic flocculation–flotation processes, such as Flocculation–Flotation-FF®, (Da Rosa and Rubio, 2005), Flocs Generator Reactor FGR®, (Carissimi and Rubio, 2005a; Carissimi et al., 2007); Bubble Accelerated Flotation-BAF® (Owen et al., 1999; Colic et al., 2001) and Air Sparged Hydrocyclone Flotation-ASH® (Miller, 2001).

Some researchers believe that the formation of aerated flocs occurred either through entrapment of small bubbles within flocs, nucleation of bubbles at floc/water interfaces, or polymer coiling as a result of salting out effects at the aqueous/air interface (Da Rosa and Rubio, 2005). The latter when flocculation is conducted under high turbulence (Da Rosa and Rubio, 2005). Some of these phenomena would be favored in flocculators operating with a plug flow regime others “ideal” (complete) mixing. Although these hypotheses are plausible, more research on a full characterization of these operating mechanisms involved in the bubble-flocs, interactions and structures is needed.

Flocs are usually characterized by structural and hydrodynamic properties such as the size, shape factor, fractal dimension, settling velocity, up-rising rate and strength to shear. The size and structure of flocs are considered important parameters for industrial processes because they are closely linked to solid/liquid separation efficiencies.
Several researchers (Haarhoff and Edzwald, 2001; Zhao, 2003; Carissimi and Rubio, 2005a; Ho and Newcombe, 2005; Jarvis et al., 2005; Li et al., 2006; Carissimi et al., 2007; Kusaka and Adachi, 2007; Nasser and James, 2007; Coufort et al., 2008) have employed several techniques to determine floc size. Among them, the most common are microscopy, capture and image analysis and transmitted light scattering. Still, visualization techniques at various length scales have been used extensively in flotation processes. More, image analysis techniques enable in situ measurements (Ducoste and Clark, 1998; Chakraborti et al., 2000; Bache and Papavasiliopoulous, 2003) with a high sampling of flocs, which results in good statistical reproducibility.

Floc size is an important parameter that can be determined from the perimeter, surface area, volume, surface-volume, Stokes, Martin and Feret equations. However, the latter approach results in a statistical diameter and provides satisfactory data in terms of particle size distributions (Allen, 2003). According to Jarvis et al. (2005), the diameters based upon two-dimensional images, such as, the projected area, Martin and Feret diameters are known as statistical values because they are an acceptable indication of the particle size distribution, should enough measurements are made.

Another important feature of flocs is the fractal geometry, which expresses how the volume is occupied by primary particles in a nominal volume containing a floc. The fractal theory is defined in linear, planar or volumetric terms (one-, two- or three-dimensional values). According to Bushell et al. (2002), the image analysis technique is versatile, mainly for determining the fractal dimension when it is applied in situ. In addition, other parameters can be obtained, namely the shape factor, which shows changes in the form of the flocs under different operational conditions (Jarvis et al., 2005).

The up-rising rate of the flocs is also a very important measure of the solid/liquid separation process kinetics and is often calculated using the Stokes equation. This theory is usually adapted for flotation by considering an incompressible and spherical solid. However, for aerated flocs which exhibit a high volume of air bubbles (and hence low flocs density); errors in the actual value of their rising rate are found. In fact, the bubbles interactions with flocs are a dynamic process leading to a continuous change in the ratio air/floc mass or flocs density.

Carissimi and Rubio (2005a) characterized aerated flocs that were generated in a flocs generator reactor (FG®) by using a floc rising cell; the flocs were observed individually over a specific time period, and the up-rising rate of a floc population was evaluated. This principle was also used by Malysa et al. (1999a,b) who showed a technique named LubaTube; this apparatus required an ascent tube, a source of light and a digital camera to capture the images of bitumen aggregates. Techniques that are similar to LubaTube are well known and have been applied in mineral processing to characterize aggregates and hydrodynamic conditions in flotation processes.

However, in addition to a better understanding of the basis of mechanisms involved in the formation of aerated flocs, the field of water and wastewater treatment still needs research on advanced techniques for accurate floc characterization and observation of multiphase systems.

The aim of this work was to develop and validate a technique named Aerated Flocs Characterization (AFC) to characterize multiphase systems in flocculation-flotation processes by using different suspension models and dissolved air flotation (DAF) or flotation with microbubbles. Main measurements on physical and interfacial properties of floc, microbubbles coalescence, and attachment onto flocs and type of solids are reported.

2. Experimental

2.1. Apparatus

The AFC was inspired by the LubaTube, which, according to Malysa et al. (1999a,b), is a technique that has been used for some years by the Syncrude Canada Research Center to study the flotation of bitumen in mineral processing. AFC is unique and has many advantages: it enables the evaluation and characterization of multiphase systems by recording the floc flows together with images of the water/flocs/air bubble systems using a coupled microscopy technique.

The AFC takes into account the principle of atmospheric pressure balance. In other words, when a cylinder filled with water is turned upside-down, opened and immersed in a water container, the water column in the cylinder can stay above the water level by equilibrating the pressure. Thus, a floc flow can rise into the column and can be well characterized (details in Section 2.3.1).

The main component of the AFC is the rising tube that consists of an acrylic circular tube with a total length of 40 cm, an inner diameter of 5 cm and a closing/opening mechanism (controlled by a remote handle) under the tube that controls the entrance of flocs into the tube (see Fig. 1). Over the tube, there is a top rectangular acrylic box (10×10×15 cm) that has three glass windows on the lateral walls (15×7 cm each) and one on top (7×7 cm). Also, this box has an opening system for cleaning.

Additionally, different acrylic tubes of 5 cm of height and 5 cm of inner diameter were placed in the bottom section. They have small end outlets having specific diameters, as 1, 2 cm and 4 cm, respectively. These diameters may be selected according to both, the required floc flow inside the tube and the sampling of varying floc sizes. The one having 1 cm of the opening diameter was used, because it sampled a satisfactory population size, and did not have wall effects.

Fig. 1 shows a schematic of the tube and a full description apparatus is given in Section 2.3.1.

2.2. Materials and reagents

Samples of kaolin (Cadam®), high-purity activated carbon (Carbomafr®) (Table 1) and Fe(OH)₃ colloidal precipitates were used as model suspensions. The selection followed mainly their differences in grain size and hydrophobicity.
The reagent FeCl₃·6H₂O (Vetec®) was used to generate the iron hydroxide colloidal precipitates. Potassium nitrate (KNO₃—Merck®) of analytical purity was used for measuring the zeta potential of the kaolin and activated carbon particles. pH adjustments were made using HNO₃ and KOH solutions. An aluminum sulfate solution (Al₂(SO₄)₃·14H₂O—Cynth®) was used as a coagulant for the kaolin particles. The high molecular weight polymers included an anionic Superfloc-A100 (Kemira®), a cationic Superfloc-C448 (Cytec®) and a non-ionic-920SH (SNF Floerger®). All of the solutions were prepared according to the manufacturer’s instructions by using water obtained from a reverse osmosis water treatment lab unit.

2.3. Methodology

2.3.1. Flocculation–flotation studies

The flocculation–flotation studies were carried out by using a dissolved air flotation (DAF) unit, composed of a 5 L pressure vessel for saturating the water at 4 atm and a 5 L flotation cell. This cell was connected by a release needle valve to depressurize the saturated water at the top window, because this is controlled and avoided by the sampling time. Consequently, the sampling time used in this work allowed to obtain a representative population, as well as, a fairly good individual positioning of the flocs at the top window.

The AFC tube was filled with water and the air was expelled by a vacuum formed when immersed at 6 cm in the suspension within the flotation cell. A digital camera (Sony Cyber-Shot DSC-S75) was placed in front of the rectangular box to take video recordings of the flocs flow; another camera (Sony Mavica MVC-DC500) was coupled to the optical microscope (Zeiss Stemi SV 11) to capture images of the multiphase system inside the tube. This camera was set up at the surface of the system with the purpose of getting qualitative data from investigations on the arrangement of the bubbles into the flocs, the bubble size distribution, the stability of the bubbles inside the flocs, the attached and the individual bubbles.

The digital cameras were used in manual mode, thus, automated features were disabled allowing a manual control of important parameters, among others, digital zoom, focus, resolution, exposure time and aperture settings. This was done to keep a suitable control and uniformity of the conditions of the image capture stage, in all studies.

An illumination device was installed opposite to the cameras to provide suitable contrast for the images.

### Table 1
Main characteristics of the kaolin and activated carbon particles.

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Kaolin</th>
<th>Carbon</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size, μm</td>
<td>0.87</td>
<td>10.9</td>
</tr>
<tr>
<td>Specific surface area, m²/g</td>
<td>17</td>
<td>703</td>
</tr>
<tr>
<td>Specific mass, g/cm³</td>
<td>2.3</td>
<td>1.6</td>
</tr>
<tr>
<td>Moisture, %</td>
<td>1.1</td>
<td>15.9</td>
</tr>
<tr>
<td>Whiteness</td>
<td>88</td>
<td>–</td>
</tr>
<tr>
<td>X-ray</td>
<td>99 % of kaolinite</td>
<td>–</td>
</tr>
</tbody>
</table>

### Fig. 2
Schematic of the Aerated Flocs Characterization (AFC) technique: (1) pressure vessel, (2) flotation cell, (3) digital camera for recording the floc flow, (4) rising tube, (5) illumination sources, (6) optical microscope, (7) digital camera for capturing the multiphase system and (8) screen to observe the resulting images.
Videos were recorded without any changes of the camera position or its focus; therefore, the established magnification was kept constant during the recordings of the flowing flocs. The manual focus operated at the maximum depth mode to give three times the magnification. Thus, the camera features were adjusted manually, as well as, the positioning of the cameras and tube. Within the tube filled with water was inserted a calibrated scale in the flocs flow zone. Thus, video recordings were captured and the images were treated in the absence of the fish eye effects. The same procedure was performed with the camera coupled in the microscope to capture images instead of video recordings. These procedures were conducted in all experiments and all videos and images were calibrated. A schematic of the apparatus is shown in Fig. 2.

Additionally to sampling using AFC, a sample of subnadir suspension from flotation cell was collected after 1 min of flotation and the residual turbidity was measured.

The same procedure using AFC was used to characterize the air bubbles, but in this case, the flotation cell was filled only with water (in the tube absence of particles). The microbubbles were injected into the flotation cell and were sampled during their uprising in the box above the tube. It is important to emphasize that for the bubble size distribution studies, the images were captured immediately, because these bubbles might coalesce on function of the time.

2.3.2. Method of image analysis

2.3.2.1. Floc characterization. All recorded videos and captured images were processed using image analysis software named Image Tool 3.0, developed by researchers from the University of Texas Health Science Center in San Antonio — UTHSCSA. Firstly, the video recordings were divided into frames over established periods \( t = 1 \text{s} \) and 25 frames were isolated; this period was sufficient to sample the flocs across the suspension models. Then, the frames were treated as follows: (a) every frame was converted to grayscale; (b) the objects in each frame were thresholded; (c) the objects were analyzed by determining their mass centers; (d) the subsequent frames (frame \( t_0 \) and frame \( t_0 + 1 \) ) were overlapped and (e) the distance between the two mass centers of the same floc was determined and the up-rising rate \( (v) \) was obtained according to Eq. (1):

\[
v = \frac{\sqrt{(x_2-x_1)^2 + (y_2-y_1)^2}}{\Delta t}
\]

where \((x_1, y_1)\) and \((x_2, y_2)\) are the coordinates of the flocs in frames \( t_0 \) and \( t_0 + 1 \), respectively, and \( \Delta t \) is the time interval between the frames.

Now, all objects (flocs) in the frames, were recognized and numbered. All objects were characterized including the mass centers parameter. When the frames were overlapped, the numbers attributed to the same object in the different frames were identified and registered manually. A list containing the pairs of attributed numbers to the same object with their correspondent mass centers was obtained. Finally, these numbers and characterization data were computed in a excel spreadsheet to make calculations of the distance between the two mass centers. The identification of the same floc was fairly and reliable and avoids errors committed by the algorithm, from the software.

Therefore, it is believed that the obtained data are precise and have satisfactory statistical reproducibility mainly because a large population of flocs (approximately 200 flocks were analyzed in each study).

Fig. 3 shows the main steps that were carried out during the image treatment to determine the up-rising rates of the flocs by measuring the displacement of flocs in 1 s.

Other characterization parameters (such as the Feret diameter, shape factor and fractal dimension) were determined using the same steps as described above. The Feret diameter \( (d_f) \) was defined as the diameter of a circle that has the same area of an irregular object and was determined using Eq. (2):

\[
d_f = \sqrt{\frac{4 \text{Area}}{\pi}}
\]

The shape factor \( (SF) \) is defined by Eq. (3) — this metric assumes values in the range of zero to one. Thus, higher values \( (SF \rightarrow 1) \) imply

![Fig. 3. Key image treatment steps to determine the up-rising rates of the flocs: (i) conversion of the frames to grayscale; (ii) identification of the objects and their mass centers; and (iii) overlap of the subsequent frames and determining the distance between two mass centers of the same floc.](image-url)
an object is a perfect circle, while lower values ($SF \to 0$) are related to a non-circular form.

$$SF = \frac{4\pi \text{Area}}{\text{perimeter}^2}$$

(3)

The fractal dimension was determined in planar terms using a two-dimensional analysis. This parameter ($D_2$) is a dimension that is related to the projected area from the primary particle mass, which constitutes a flocc in a circle of radius $r$, according to Eq. (4) (Harrison, 1995; Chakraborti et al., 2000). The average $D_2$ values are in the range of zero to two; thus, lower values ($D_2 \to 0$) describe a structure that is open, large, highly branched and loosely bound while higher values ($D_2 \to 2$) imply a structure that is more compact and densely packed.

$$A \propto r^{D_2}$$

(4)

where $A$ is the sum of the areas of all of the primary particles in a circle of radius $r$. Thus, $D_2$ is the slope of the plot ($\log A$) versus ($\log r$).

The effective flocc density ($\rho_f$) was determined according to the modeling by Haarhoff and Edzwald (2001), who used it to characterize the bubble–floc aggregates in a dissolved air flotation system.

The parameters as fractal dimension, shape factor and density were expressed as mean values (arithmetic mean data).

2.3.2.2. Bubble characterization. Bubble images were treated using the same procedure used to characterize the flocs. However, in this case, the longer diameter (major axis length) was determined, because when the images receive the threshold treatment, the bubbles appear with both an irregular form and small white areas that are caused by light reflection onto the bubbles during the studies (see Fig. 4).

Another important fact was the elimination of those objects that constituted both touching bubbles and bubbles on the frame border. Fig. 4 shows the main steps of this characterization.

3. Results and discussion

3.1. Flocculation–flotation studies

3.1.1. Activated carbon particles

The flocculation–flotation studies of the activated carbon particles, using the anionic polymer A100 showed satisfactory performance in terms of particles removal (99%) with low residual turbidity (8.3 NTU) in the treated liquid. Fig. 5 shows an up-rising rate distribution in the range of 25–85 m/h. These data show that the predominant rising velocity was between 55 and 65 m/h while some faster flocs (85 m/h) appeared to indicate the formation of some aerated flocs.

In this aggregate flow, flocs had a wide size distribution in the range of 0.7–3.3 mm (Fig. 6). Also, the majority of flocs (>80%) were smaller than 1.9 mm while the bigger flocs 2.3–3.3 mm, accounted for just 10% of the population. This indicates that this polymer induced a
good growth for the primary flocs. The shape factor (0.8), fractal dimension (1.2) and density (0.98 g/cm³) of the structures were characterized; the structures held many particles with a medium compactness that resulted in light, open structures.

The microscopic analysis of this multiphase system showed plenty of microbubbles in all structures of activated carbon. This fact is due to hydrophobic interactions between carbon particles and microbubbles; the latter remaining stable inside the structure with no bubble coalescence being observed. However, the faster flocs (85 m/h) appear to be due to the great number of other, but entrapped bubbles.

The microscopic analysis of the multiphase system formed by water/bubbles/activated carbon containing plenty of microbubbles and large bubbles attached.

3.1.2. Kaolin particles

Flocculation–flotation studies of the kaolin particles using the non-ionic polymer 920SH was very effective yielding low residual turbidity (24.8 NTU) in the treated liquid. The kaolin flocs showed an up-rising rate distribution in the range of 35–105 m/h. The predominant rising velocity was between 65 and 75 m/h (Fig. 8); and, some (5%), rising very fast (85–105 m/h).

Here, the floc population had a size distribution in the range of 0.5–3.3 mm (Fig. 9) while the majority of the flocs (80%) were smaller than 1.3 mm. The large flocs only accounted for a small population; however, they were faster indicating, again, the formation of aeroflocs. The mean values of the shape factor, fractal dimension and density were 0.9, 1.0 and 0.97 g/cm³, respectively; this corresponds to light, open structures with high particle concentration and medium compactness values.

The microscopic analysis of the multiphase system formed by water/bubbles/kaolin flocs suggested that some flocs had several air bubbles in their floc structures, while others only included a few bubbles. This result was the opposite of activated carbon model, where the flocs exhibited plenty of bubbles.

This difference can be explained by the chemical–physical properties of these particles, because carbon particles are more hydrophobic than kaolin and, therefore, they have more of an affinity for air bubbles (hydrophobic interactions).

Although some kaolin flocs exhibited fewer bubbles (less hydrophobic mineral), the high up-rising metrics persisted due to the presence of large bubbles in these so-called aeroflocs. This is in agreement with Carissimi and Rubio (2005a) who reported a relationship between the theoretical data of equivalent diameter of bubbles and the up-rising rate. These authors also argued that large bubbles are responsible by high up-rising rates and it would explain the term aerated flocs. Currently, this hypothesis is confirmed using AFC technique whose obtained images showed clearly the presence of large bubbles in the floc structure.

3.1.3. Fe(OH)₃ particles

The flocculation–flotation studies of Fe(OH)₃ colloidal particles that used the cationic polymer C448 showed satisfactory performance
in terms of particle removal (95%) and, therefore, low residual turbidity (0.5 NTU) in the treated liquid. Fig. 11 shows the up-rising rate distribution in the range of 45–145 m/h. The predominant rising velocity was between 65 and 85 m/h while some very fast flocs (7%) with a velocity, in the range of 125–145 m/h, were also observed.

This floc population showed a size distribution in the range of 0.5–2.9 mm (Fig. 12). Most of the flocs (70%) were smaller than 1.1 mm while the large flocs were definitely in a small number, but still rose very fast. Also, parameters such as the shape factor, fractal dimension and density were 0.7, 0.8 and 0.95 g/cm³, respectively, indicate a very open floc structure.

The microscopic analysis of this multiphase system showed a great number of bubbles in all the flocs observed (Fig. 13). The structures seemed very open, i.e., very filamentous, with many empty spaces and loose bonds.

Some flocs had many large bubbles and yielding faster up-rising values. This fact may be due to the very open and porous floc structure which allows a very tight packing of many bubbles that grew more readily than those distant bubbles in the compact flocs.

Fig. 13 shows clearly an aerated floc of iron hydroxide, notably structures having large bubbles responsible for the high up-rising rates to these flocs.

Another important feature is that results and discussions, presented in this work, confirmed that fractal dimension appears to be not relevant to the up rise rates, a fact which is in agreement with Haarhoff and Edzwald (2001). This parameter is described in this work with the purpose of showing that it is possible to obtain fractal dimension values using this technique, another alternative technical contribution.

3.2. Bubble characterization

The bubble characterization suggested a size distribution in the range of 10–230 μm with a mean diameter of 73 μm (Fig. 14). But, the majority (70%) of bubbles was between 50 and 90 μm in diameter while 83% were smaller than 110 μm. These data are consistent with the results of Rodrigues and Rubio (2003), who developed the technique named LTM-BSizer to measure the bubble size and the bubbles size distribution in the context of dissolved air flotation (DAF). Researchers who used the same experimental conditions as in this manuscript have reported a mean bubble size of 65 μm.

Finally, it is believed that AFC technique may be utilized to study other different factors apart from those aiming in this work; namely the reagent chemistry effects, ionic strength, and presence of ultrafines, bubble–particle attachment rates, in general flocculation–flotation processes.
4. Conclusions

A technique to characterize multiphase systems, named *Aerated Floc Characterization* (AFC), was developed and validated in flocculation–flotation systems of different suspension models. The approach enables the sampling and recording of floc flows to better understand the multiphase system, which is formed by water/floc/air bubbles. The technique allowed to perform a detailed evaluation of aerated flocs by measuring parameters, such as the size, shape factor, up-rising rate, fractal dimension and density of the flocs, with a high statistical reproducibility. The AFC allowed both the characterization of microbubbles that were generated in a Dissolved Air Flotation (DAF) unit, and the calculation of a relationship between the bubble size and the up-rising rates of flocs and bubbles. AFC allowed the identification of bubbles (various sizes), bubbles attached and entrapped in the so-called aerated flocs and appears to have a good potential for the understanding of floc-flotation, a rapid separation of flocs from water and for measurements of bubbles–particles attachment rates.

**Abbreviations and units**

- $V$: volume
- $T$: time
- $\Delta t$: time interval
- $D$: distance
- $d_f$: Feret diameter
- $SF$: shape factor
- $D_2$: fractal dimension
- $A$: area
- $r$: radius
- $P_r$: floc density
- $m$: mass
- $T_I$: mean value of the initial turbidity
- $\left[\right]$ concentration
- $P_s$: saturation pressure
- NTU: Nephelometric Turbidity Units
- AFC: Aerated Flocs Characterization
- DAF: Dissolved Air Flotation

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**Fig. 13.** Aerated floc of iron hydroxide colloidal precipitates — microphotographs showing details of large bubbles entrapped in the floc structure.

**Fig. 14.** Bubbles injected into the flocculation–flotation studies and their size distribution. Conditions: $V$ of 2 L; $P_s$ of 4 atm; and recycle ratio of 30%. Statistical data: mean of 73 μm; minimum of 8 μm and maximum of 222 μm.