# MEASUREMENT AND ANALYSIS OF MICRO-BUBBLES PRODUCED IN ELECTROFLOTATION[1]

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**Abstract**

Bubble sizer distribution is of great importance in flotation because smaller bubbles cause an increase in interfacial area and hence and increase in the efficiency of the separation process. It is recognized the ability of electroflotation to create very fine bubble, which are known to improve flotation performance of fine particles. In this work, an electroflotation cell was designed and constructed in acrylic with stainless steel cathode and Ti/$RuO_2$ mesh as anode. The measurement and analysis of bubbles generated in different electrolyte concentrations have been studied at different applied currents. Bubble size distribution (BSD) was measured by using two methods: image analysis (high-speed digital camera) and, the laser diffraction technique. From the analysis it was found that, current density and pH influence the process. In the range used, higher current densities allow a larger number of bubbles to be obtained. Higher pH values also enhance the production of bubbles. Both, image analysis and the laser diffraction technique allow assessing bubble size in the range of current densities studied. However, there is difference between the values obtained in each method. The Sauter mean bubble diameter obtained ($H_2/O_2$) was 82 µm to 92 µm with image analysis and 40 µm to 58 µm using laser diffraction method. Results obtained are in good correlation with values reported in literature.

**Key words**: Electroflotation; Bubble sizer; Micro-bubbles; Fine flotation.

## MEDIÇÃO E ANÁLISE DE MICROBOLHAS GERADAS NA ELETROFLOTAÇÃO

**Resumo**

A determinação de tamanho de bolhas é de grande importância no processo de flotação, bolhas menores tem uma maior área superficial, consequentemente é possível ter um aumento na eficiência no processo de separação. É conhecida também a capacidade do processo de eletroflotação para produzir microbolhas capazes de melhorar a eficiência na flotação de partículas finas. Neste trabalho foi projetada e construída uma célula de eletroflotação de material acrílico, usando aço inox como cátodo e uma malha de Ti/$TuO_2$ como ánodo. A medição e analise de bolhas geradas foi realizada em diferentes valores de pH e a diferentes densidades de corrente, logo a distribuição de tamanho de bolha foi medida usando dois métodos: Analises de imagens (usando câmara digital de alta velocidade) e a técnica de difração laser. Os resultados indicaram que tanto a densidade de corrente quanto o pH foram as variáveis mais influentes no processo, maiores densidades de corrente e pH na faixa alcalina produz um maior numero de bolhas. É possível avaliar a distribuição de tamanho de bolha por ambas técnicas.Embora existem diferencias entre os valores obtidos em cada método. Assim, o tamanho médio de Sauter obtido usando a técnica de analise de imagens foi de 82-92 um enquanto que usando a difração de raios laser foi de 40-58 um. Os resultados obtidos tem correlação com os valores apresentados na literatura.

**Palavras-chave**: Eletroflotação; Tamanho de bolha; Micro-bolha; Glotação de finos.

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## 1 INTRODUCTION

Electroflotation consists of the production of bubbles through electrolytic processes. Electrodes used for electroflotation consist of inert materials (e.g. platinum, graphite, titanium or stainless steel) that can be used as anode or cathode depending on the requirements in each case. Typically, when electroflotation is applied in an aqueous media, electrolysis of water produces tiny bubbles of oxygen and hydrogen through the reactions:

$$\text{Cathode} \quad 2H_2O + 2e^- \leftrightarrow 2OH^- + H_2(g) \qquad (1)$$

$$\text{Anode} \quad H_2O \leftrightarrow 2H^+ + \frac{1}{2}O_2(g) + 2e^- \qquad (2)$$

These electrolytic bubbles, with an average diameter of 20 µm to 40 µm[1-3] are smaller in size than others methods for bubble generations, and it is most likely the lifting capacity of such small bubbles is sufficient for the fine particle be floated.
Electroflotation of minerals fines have been extensively studied. Raju and Khangaonkar[4] reported 74%–81% recovery of 4 µm chalcopyrite. Ketkar, Mallikarjunan e Venkatachalam[5] reported more than 60% recovery of +4–10 µm quartz with hydrogen bubbles. Llerena, Ho and Piron[6] reported almost 100% recovery of 25 µm sphalerite. Han, Kim e Ahn[7] used electroflotation to generate very fine bubbles with 27 µm mean diameter to obtain 98% recovery of 28 µm flocculated kaolin particles. Most recently, Sarkar, Donne and Evans[8] obtained more than 70% recovery of 13 µm silica particles with hydrogen bubbles less than 40 µm diameter. Many of the studies listed have reported that the recovery of particles in the diameter range of 1 µm to 30 µm is increased with decreasing bubble size, which is largely the result of the increased collision efficiency between the fines particles and the bubbles.
Electrolytic bubble characteristics are dependent on the physical processe of bubble evolution on the electrodes surface and in the bulk electrolyte. The product of the water splitting reaction via electrolysis is in the form of dissolved gas in the electrolyte. When its concentration exceeds the supersaturation limits, a bubble is nucleated. Then the nucleated bubble grows and coalesces with its bubble neighbors on the electrode's surface. When they reach a certain size, they depart from the surface.
Bubble nucleation in the vicinity of gas evolving electrodes requires much less dissolved gas supersaturation than the concentration for nucleation in the bulk. Thus, it is reasonable to conclude that the surface decreases the energy barrier for the nucleation process.[9] In electroflotation the complexity of the nucleation process, both at the cathode surface and in the bulk, is increased due to the presence of solids which themselves can act as nucleation sites. When these sites are very close to each other, the surrounding bubbles affect every individual bubble's growth and even coalesce with each other.
The size of bubbles produced by electroflotation is influenced by a number of factors. Such as, the type of electrode material and its position in the electromotive series.[10] It is a function of pH, with hydrogen bubbles being larger in an acidic medium compared to a neutral or alkaline medium. Similarly, oxygen bubbles attain a minimum size in acidic medium and increase in diameter with increased pH.[11] The detachment size of bubble also depends on the curvature of the electrode surface.[12] For a constant current density and pH, detached bubble diameter has been reported to decrease with decreasing wire (electrode).[13]

Several methods have been developed to measure the size of bubbles. Rowe and Everett[14] employed X-ray. Chiba, Terashima e Kobayashi[15] and Saxena, Martur and Sharma[16] used cinematography techniques combined with X-ray. Atkinson and Clark[17] used pressure probes and Sung and Burgess[18] measured with a laser. Other methods reported in the field of mining and environmental engineering are: Image analysis (photographic, digital images and videos) by Unno and Inoue,[19] Ahmed and Jameson;[20] Electroresistivity by Han, Park and Yu;[21] Optical by Biswal, Reddy and Bhaumik;[22] Optical (optical-sensors) by Han, Park and Yu;[21] Porous plate by Randall et al.,[23] Tucker et al.,[24] Gorain, Franzidis and Manlapig[25] and O_Connor, Randall and Goodall;[26] Drift flux analysis by Dobby, Yianatos and Finch,[27] Filippov, Joussement and Houot,[28] Yoon[29] and the acoustic bubble sizer (Dynaflow, Inc.) Wu and Chahine.[30]
Bubble size characterisation using optical means is by far the most widely employed technique. Often times depending on the size and number of bubbles in question as well as the quality of optical device, the optical method can be both painstaking and time consuming to undertake and its accuracy is a function of factors such as light and medium clarity as well as the software for bubble analyses. These factors if not properly addressed can give rise to errors such as underpredicting or over-predicting the bubble diameter particularly in high bubble flux conditions and turbid media.
The laser diffraction, or low angle laser light scattering (Lalls) technique is classified as non-destructive and non-intrusive and relies on the fact that laser diffraction angle is inversely proportional to particle size.[31,32] It has a large size range from 0.01 µm to 3.500 µm, depending on the equipment layout. A typical system consists of a He-Ne laser (light source of fixed wavelength 632.8 nm) and suitable detectors to measure the light scattering pattern, and a PC for signal processing and results output.[32]
Laser diffraction results are calculated as volume-equivalent spherical diameter (ESD). As the micro-bubbles (with diameter less than 100 µm) are considered rigid spheres,[33] measurements are not subject to stereological conversions due its shape,[34] as most solid particles are. In fact, the laser diffraction technique was previously used on micro-bubble size measurements of colloidal gas aphrons (CGA), type of foam usual on separation process and DAF process.[35-37]
This work presents two methodology for characterization of micro-bubble size distribution in the EF process by the laser diffraction technique and optical techniques already known. The size and number of bubbles is a vital operating and control variable and must be appropriate for effective bubble-particle contact. Characterising the bubbles generated from a flotation unit is a necessary first step and was undertaken prior to recovery of fines. The influence of key parameters on the micro-bubble size, like pH and current density were investigated.

## 2 MATERIALS AND METHODS

### 2.1 Electroflotation Cell e Electrodes

The electroflotation cell was constructed of acrylic material with 1 L of capacity operated in batch mode. A polished stainless steel with 8.5 cm of diameter and total surface area of 56.7 cm$^2$ was used as cathode. It was placed in the bottom of the cell and a Ti/$RuO_2$ mesh supplied by De Nora was used as the anode (3 mm above the cathode). This configuration has been chosen to measure both the hydrogen and oxygen bubbles produced and at the same time. The electrical current was applied using a TectroL mod.–TCA-30-10XR1A DC power supply with a maximum current rating of 10 A at an open circuit potential of 30 V. To assess the effect of pH, current density was held constant (49.4 mA/cm$^2$). When, the current density effect was assess, the pH was adjusted to 7.0. All experiments were performed at an ionic strength of 0.1 M NaCl.

## 2.2 Electroflotation Micro-bubble Size Distribution Assessment Through Image Analysis Cell

These experiments were performed using The Anglo Platinum Bubble Sizer technology. Figure 1 shows the bubble size equipment. It comprises of a sampling tube attached to the bottom of a sealed viewing chamber. Bubbles from the pulp phase within the flotation cell travel in the sampling tube under non iso-kinetic conditions. The viewing chamber is made of plastic PVC with a single reinforced glass window. A lighting array (LED lights within the viewing chamber) is used to ensure image contrast. The chamber is sloped (angle 15%) to spread the bubbles into a single layer to limit overlap and provide an unambiguous plane of focus. The sample of bubbles is photographed with a digital still camera and specialist image analysis software processes the images to derive the bubble size distribution. Pictures were taken with 3.8x optical zoom. So, the final results were corrected properly.

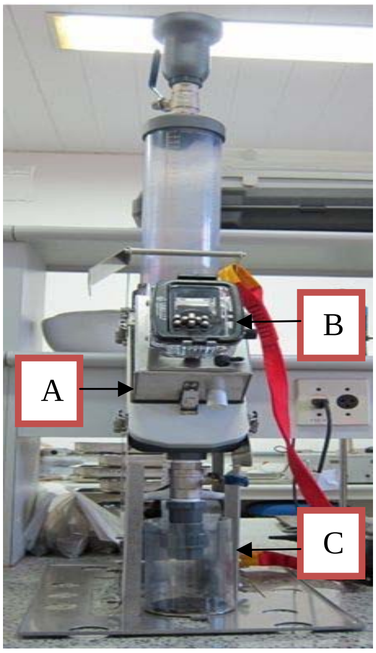


**Figure 1**. (a) The anglo platinum bubble sizer device; (b) camera; and (c) EF. cell

## 2.3 Electroflotation Micro-bubble Size Distribution Assessment Through Laser Diffraction

Also, the size distribution of micro-bubbles generated for electroflotation experiments (EF) was determined using Mastersizer 2000SM equipment from Malvern Instruments, UK, through light scattering. This instrument is capable of analysis in the range from 0.1 µm to 2,000 µm, and calculations can be accomplished through Mie's complete theory. The equipments internal model checking procedure allowed for weighted residuals ranging from 0.23% to 0.54%, below 1% considered the upper limit for good fitting.[37] A picture in Figure 2 shows the experimental setup. Pumping speed was kept low (1,000 rpm), to avoid undesired bubble formation through atmospheric air entrance. The amount of micro-bubbles in suspension was adjusted by current supply, for an obscuration between 10% and 20%. Analysis was started immediately, to avoid bubble coalescence or collapse imposed by shear. When more measurements are performed in sequence, the bubble size distribution can be shifted to the right side (larger size).

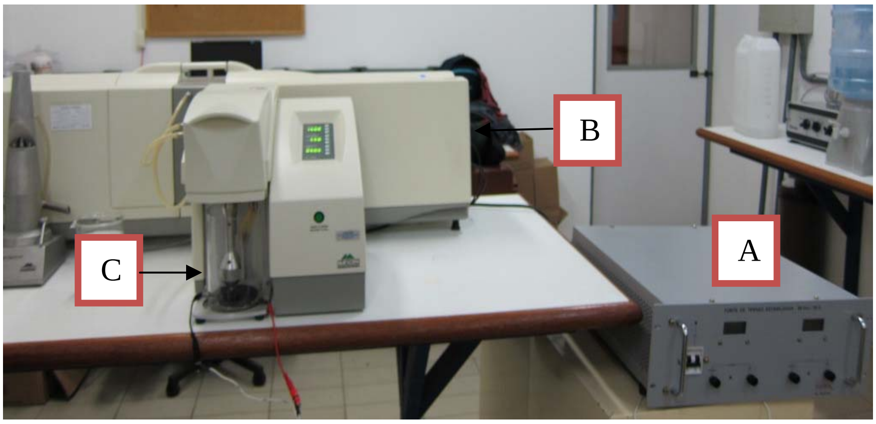


**Figure 2.** Experimental set-up using laser diffraction. (a) DC power supply; (b) Metasizer 2000 equipment; and (c) Electroflotation cell.

## 3 RESULTS AND DISCUSSION

### 3.1 Bubble Visualization and Analysis

A digital image processed by specialist image analysis software is shown in Figure 3. It can be seen in the image that bubbles were detected by three different colors (red, yellow and green) only the green color was consider bubble by image analysis software. To evaluate each point a series of 5 consecutive photographs was taken and the cumulative average diameter and number of bubbles was evaluated. To laser diffraction measurements were realized 3 times. Typical result analysis reports are shown in Figures 4a and 4b from image analysis and laser diffraction respectively.

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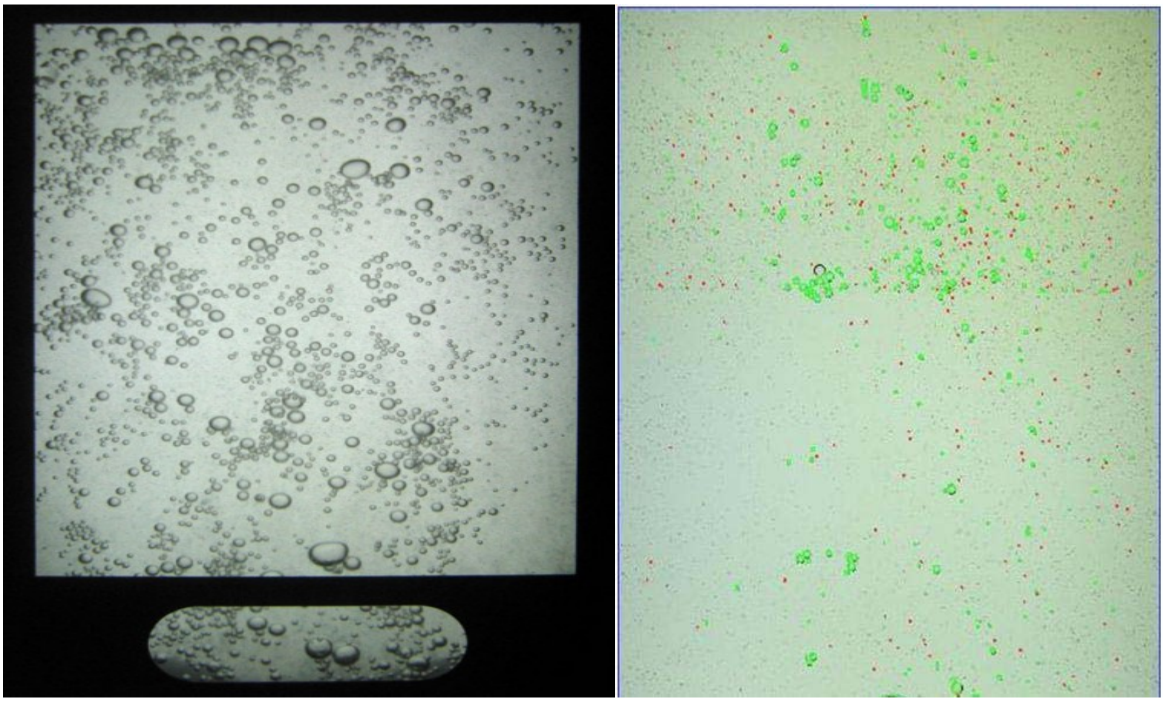

**Figure 3**. A digital image (a) before and (b) after processing by software with 3.8x optical zoom.

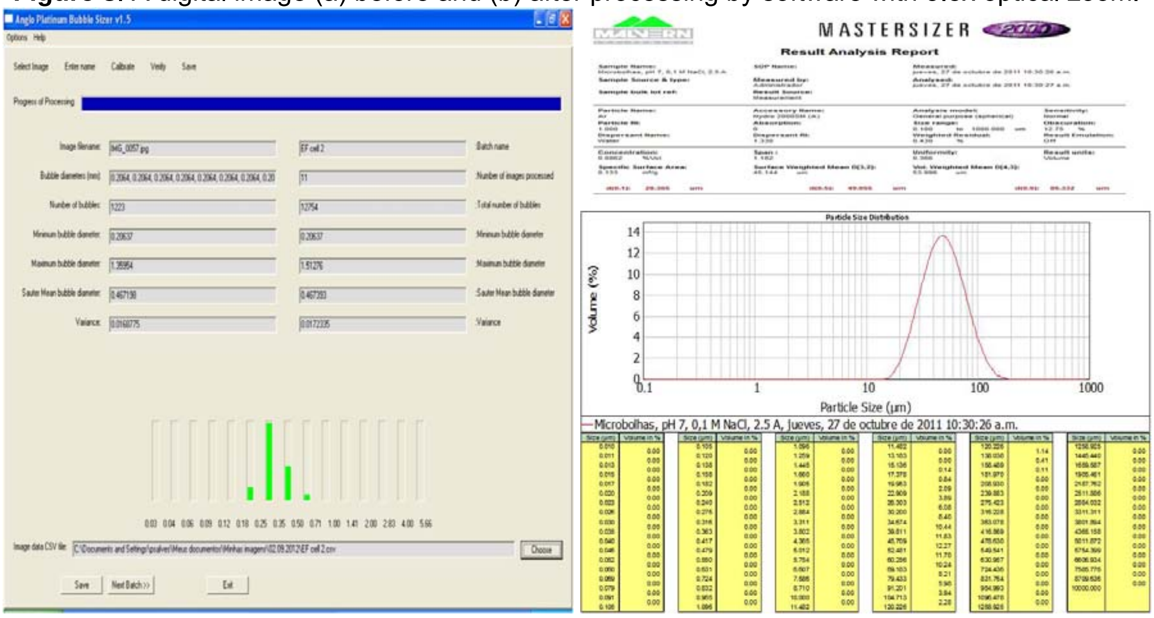


**Figure 4**. Result analysis report (a) image analysis; and (b) laser diffraction.

### 3.2 Effect of Current Density

The mean bubble measurements as a function of current density (from 8 mA/cm$^2$ to 100 mA/cm$^2$) from image analysis and laser diffraction are shown in Figures 5a and 6a. The mean diameter of the distribution from image analysis and laser diffraction are 86 and 49 um respectively. Figure 5a illustrates that the mean bubble diameter increases slightly with the current density. The formation of a huge number of bubbles can promote collisions and hence can lead to an improvement in the coalescence process.
At current densities upper of 70 mA/cm$^2$ the dynamics of bubble evolution is very variable. Despite the measurement difficulties, the data illustrates the order of magnitude of the bubble sizes and shows the trend of bubble size with current

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density. For current density lower than 100 mA/cm$^2$. The literatures reported that mass transfer is the net determining of bubble growth (some of the dissolved gas is transferred to the bubble and rest is diffused to the bulk electrolyte).[9]

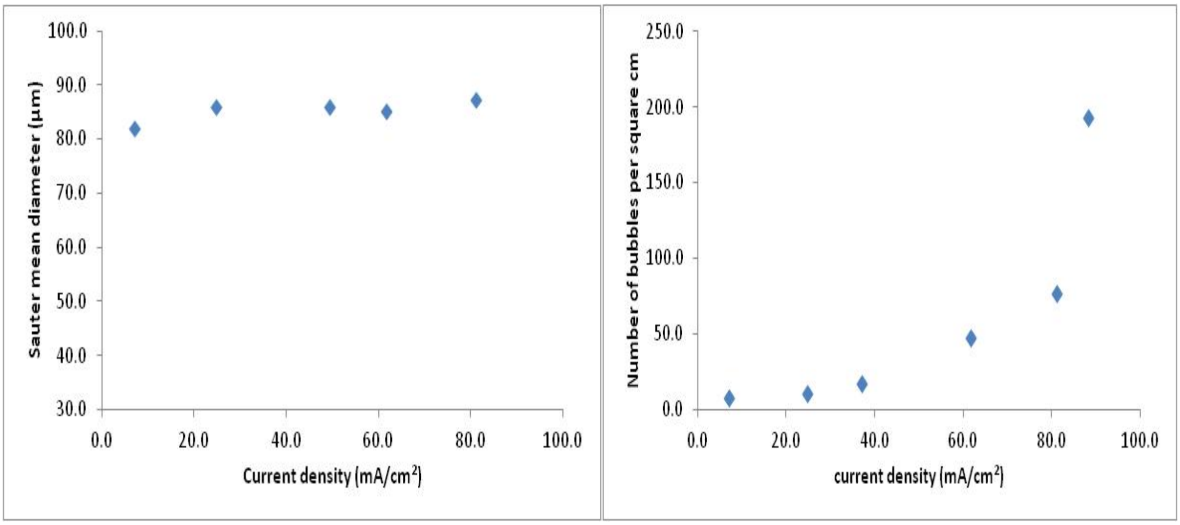


**Figure 5**. Effect of current density on the size distribution of bubbles and production (hydrogen and oxygen) through image analysis (a) Sauter mean bubble diameter; (b) production of bubbles.

It can be seen in Figure 6a that measurements bubble sizer distribution through laser diffraction were smaller than measurements from imagine analysis. Moreover, the bubble diameter was relatively constant over the entire current density range investigated. Both observations are consistent with previous studies.[9]
The data plotted in Figures 5a and 6a are not complete for higher current density at upper positions due to over saturation of bubble concentration in the electroflotation cell.
The validity of quantitative data produced from both methods is limited for a high current density. The measurements can only be reliably made for current densities lower than 100 mA/cm$^2$, due to bubble density. The image analysis method also requires absolute reliability in software to differentiate bubbles in a crowded bubble population. In addition, the number of measured bubbles may not represent the whole bubble population on the electrode surface.

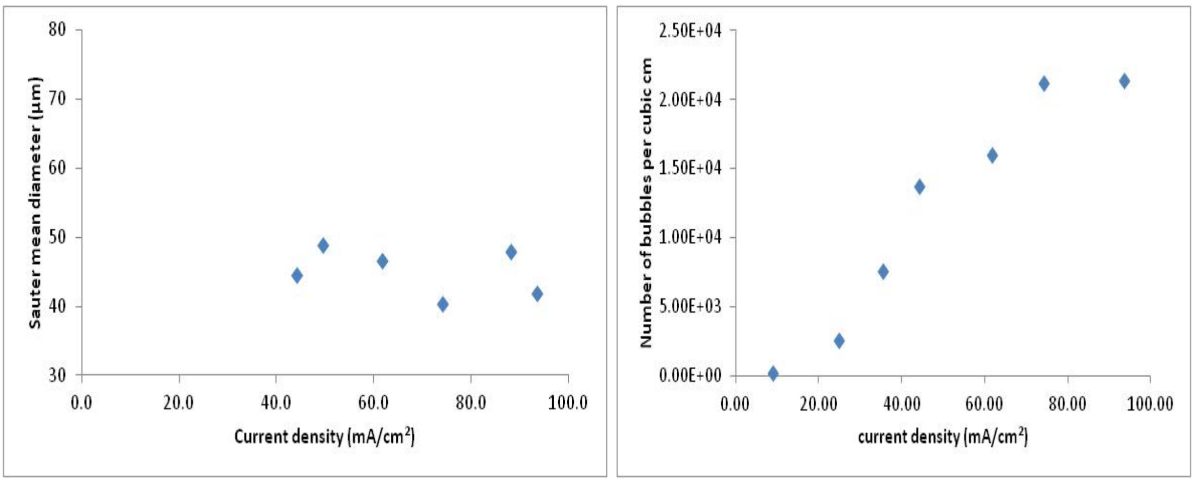


**Figure 6**. Effect of current density on the production and size distribution of bubbles (hydrogen and oxygen) through laser diffraction (a) Sauter mean bubble diameter; (b) production of bubbles.

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The effect of current density on the production of bubbles is clear for both methods (Figures 5b and 6b): the higher the current density the higher the production of bubbles during electrolysis.

### 3.3 Effect of pH

In order to investigate the effect of pH on bubble size, the pH of deionised water in the 1 liter batch electroflotation cell was varied from 2 to 12 using 0.1 M sulphuric acid or 0.1 M sodium hydroxide. It can be seen from Figures 7a and 8a that solution pH had very little effect on the mean bubble size produced. If (natural) solution pH was reduced to 2 using sulphuric acid and then increased to 4, 6, 7, 8, 10 and 12, incrementally using sodium hydroxide, the bubble size was found to have almost the same values. This indicates that pH has little effect on mean bubble size, and that any effect measured will result from the change in ionic strength of the solution. This might indicate that findings previously published which showed that bubble size increased with increasing pH were probably in error.[9] The effects of pH on bubble production are shown in Figures 7b and 8b. It can be seen that an increase in pH causes a larger number of bubbles. So, the pH of the electrolyte medium is of great importance as it can affect the volume of gas bubbles produced in the hydrogen and oxygen evolution in electroflotation.

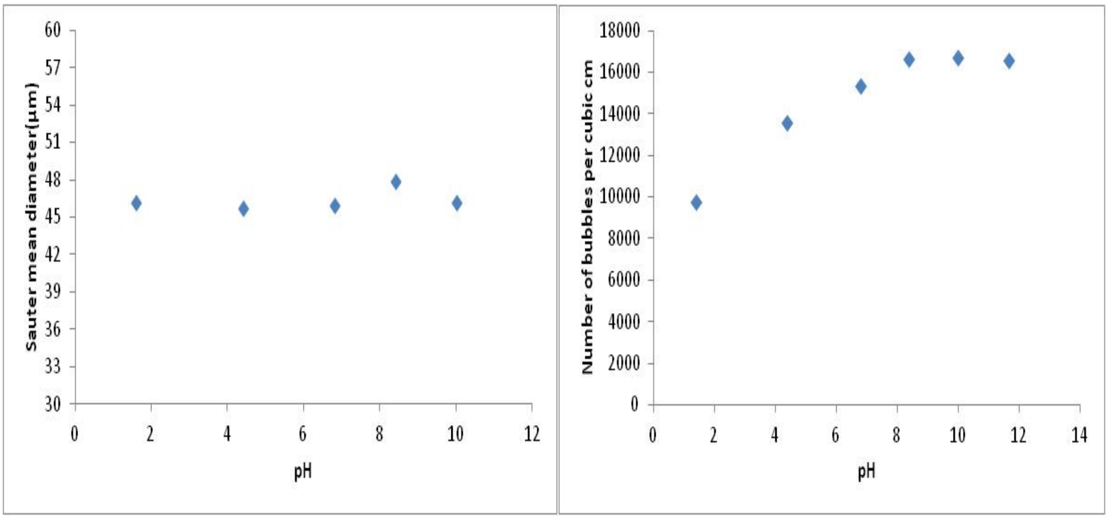


**Figure 7**. Effect of pH on production and size distribution of bubbles ( hydrogen and oxygen) through laser diffraction: (a) size distribution; (b) production of bubbles.

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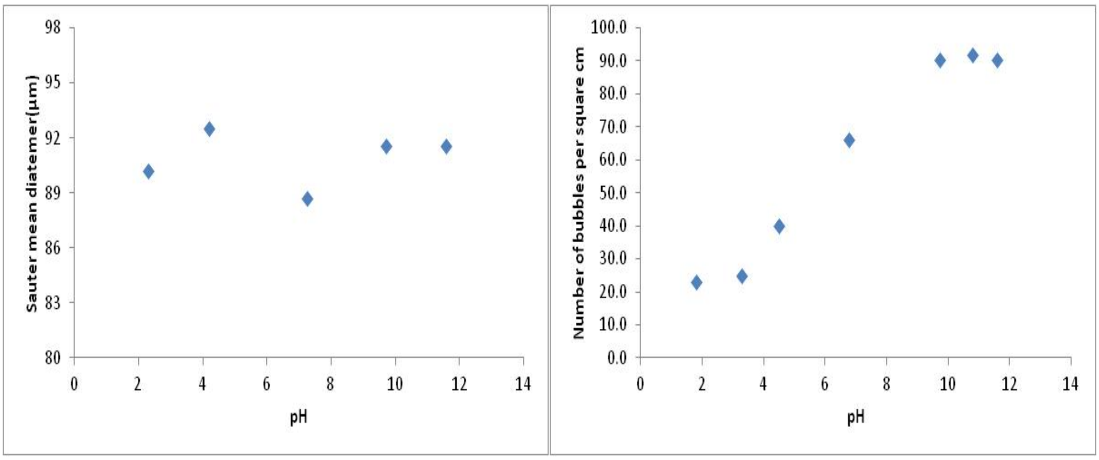

**Figure 8**. Effect of pH on production and size distribution of bubbles ( hydrogen and oxygen) through Image analysis: (a) size distribution; (b) production of bubbles.

## 4 CONCLUSION

Current density and pH are very important parameters in the electrolytic production of bubbles. Within the range studied (current densities lower than 100 mA/cm$^2$, high current densities and pH values promote the formation of larger numbers of bubbles. However, the mean bubble sizer distribution remained almost constant. This work demonstrates that is possible to obtain bubble sizer distribution from image analysis and laser diffraction methods. The two methods exhibited differences in result which may reflect the fact that there are differences in methodologies. So, comparison between these methods was not possible.

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