



# Electrochemical production of colloidal sulphur by reduction of sulphur dioxide using a parallel plate reactor with convergent flow



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## ARTICLE INFO

### Article history:

Received 25 February 2015  
Received in revised form 18 May 2015  
Accepted 20 May 2015  
Available online 30 May 2015

### Keywords:

colloidal sulphur  
sulphur dioxide  
convergent flow  
electrochemical reactor  
sulphur production

## ABSTRACT

A parallel-plate electrochemical reactor with a double convergent flow at the cathodic and also at the anodic compartment is analysed for the production of colloidal sulphur by reduction of sulphur dioxide. A reactor with convergent flow was adopted in order to enhance the mass-transfer conditions along the electrode length. A mathematical treatment is presented for reactors with convergent flow based on the plug flow model, which can predict the maximum current. However, at higher times the cathode is blocked by the deposited sulphur decreasing the current. The best working conditions were achieved with a polished cathode working at a potential of  $-0.7\text{ V}$  against SCE for a volumetric flow rate of  $8.33 \times 10^{-6}\text{ m}^3\text{ s}^{-1}$  obtaining  $19.6\text{ kg m}^{-3}\text{ h}^{-1}$  as the maximum value of space time yield, for which the electrical parameters were  $2.6\text{ V}$  average cell voltage,  $22\text{ kWh kg}^{-1}$  specific energy consumption and  $40\%$  current efficiency. The colloidal sulphur presents particles of spheroidal shape with an average diameter of  $1.85\text{ }\mu\text{m}$ .

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## 1. Introduction

Finely divided elemental sulphur suspended in water, called as colloidal sulphur, has been used traditionally as a fungicide [1,2] and pesticide. Sulphur, generally applied by spraying, has the advantage of not being consumed by the plant; residues are washed off by rain and act as a nutrient in the soil being used as a fertilizer [3]. Other users of elemental sulphur are the pharmaceutical [4], cosmetics, photographic, soap, and dye industries [5]. Nanosize sulphur particles are useful for modification of carbon nano tubes [6,7], and synthesis of nano composites for lithium batteries [8,9]. Likewise, the shape and size of sulphur particles are important factors affecting their properties and applications.

The production of colloidal sulphur can be carried out by means of physical, chemical and electrochemical procedures. The physical methods include:

- (i) solvent/non-solvent precipitation process. La Mer and Dinegar [10] examined the addition of water to ethanol or acetone solutions of sulphur and Shamsipur *et al.* [11] used the systems carbon disulfide / acetone at room temperature and hot-ethanol / water.

- (ii) Grinding of a sulphur slurry in a ball mill in presence of surfactants [12].
- (iii) Use of amino acids as modifiers to modulate nanoparticles [13].

Chemical procedures were also proposed:

- (i) Disproportion of thiosulphate in inorganic [14] or organic acid media assisted by surfactants [15].
- (ii) Oxidation of sodium sulphide by air [16] or using Fe(III) as oxidizing agent in presence of chelates [17].
- (iii) Reaction between sodium polysulfide and hydrochloric acid in a reverse microemulsions system [18].

Electrochemical proposals were also examined by the production of colloidal sulphur:

- (i) Sodium thiosulfate is first reduced at the surface of a cathode and, in the next step, the produced sulphide ions is oxidized to elemental sulphur at the anode [11].
- (ii) Oxidation of hydrogen sulphide ions to elemental sulphur at the anode coupled either to hydrogen evolution at the cathode or to oxygen reduction in a gas diffusion electrode giving a fuel cell arrangement [19].
- (iii) Elemental sulphur is generated by reduction of sulphur dioxide at the cathode of an electrochemical reactor [20].

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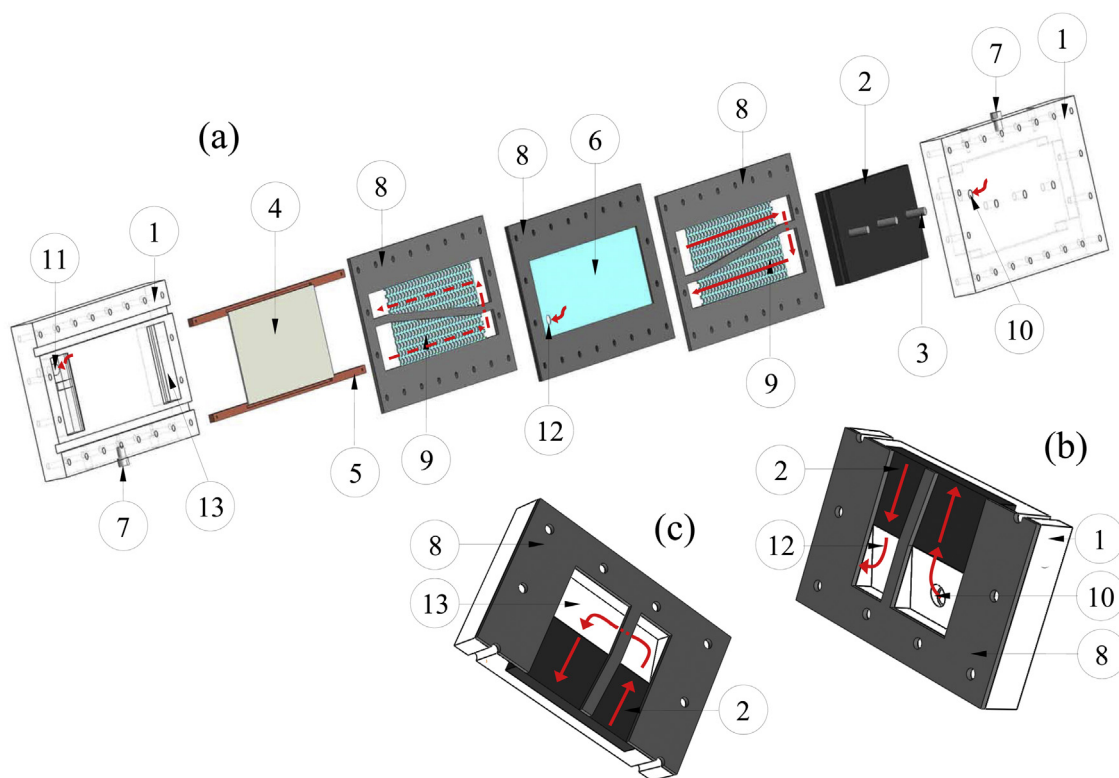
This strategy allows simultaneously the removal of the pollutant sulphur dioxide and its conversion to a useful chemical product.

Studies with a rotating disc electrode [20] demonstrated that: i) for a 316L stainless steel cathode at a potential of  $-0.5$  V, against a saturated calomel electrode (SCE), the sulphur dioxide reduction occurs at limiting current conditions, ii) the high current density and the narrow range of potentials where the limiting current is observed suggest that this reaction can be carried out in reactors with a bi-dimensional cathode and iii) at potentials more negative than  $-0.5$  V, hydrogen evolution also takes place and the current efficiency for sulphur production diminishes. On the other hand, hydrogen evolution has two beneficial effects given by the increase of the mass-transfer by bubble induced convection and assists in dislodging the sulphur layer from the cathode.

Electrochemical reactors with parallel-plate electrodes are the most widespread configuration used in the industrial practice and its versatility has been fully demonstrated [21]. Moreover, when the reaction of interest is under mass-transfer control a strategy to enhance the reactor performance is the use of a convergent channel [22], where a continuous reduction in the cross-section area for the solution flow is used as a means of improving mass-transfer in a parallel plate electrochemical reactor. Depending on the operating conditions, the convergent flow increases the mean mass-transfer coefficient by 10–60% and mass-transfer distribution under laminar flow becomes more uniform. The aim of this paper is to examine the behaviour of a parallel-plate electrochemical reactor with a double convergent channel for the electro-synthesis of colloidal sulphur by reduction of sulphur dioxide operating at limiting current conditions.

## 2. Experimental

The reactor was made of acrylic material, 100 mm wide and 270 mm long as inner dimensions, arranged in a filter press configuration. The complete experimental arrangement is schematically depicted in Fig. 1. A graphite sheet, grade 6503 Carbone Lorraine (Mersen, Buenos Aires, Argentina), 10 mm thick, was used as anode and it was electrically fed in its backside by means of three threaded screws of stainless steel. The graphite anode supports a GF-S4 graphite felt, 0.3 mm thick, supplied by The Electrosynthesis Co., Inc. (Lancaster, NY, USA) in order to increase the anodic surface area. The cathode was a sheet of 316 stainless steel, 0.5 mm thick, electrically connected along its two lateral sides by means of copper current feeders, which were joined to the dc power supply at both ends to ensure isopotentiality of the metal phase. The geometric surface area of the cathode was  $235.7$  cm<sup>2</sup>. The anodic and cathodic compartments were separated by a sheet of microporous polyethylene (Daramic Industrial CL, Charlotte, NC, USA), 0.4 mm backweb thickness and 58% porosity, in order to avoid the deactivation of the anode by the elemental sulphur cathodically produced [20]. Each compartment presents at its ends chambers of a triangular cross-sectional area, which were filled with expanded plastic meshes to homogenize the flow. Fig. 1 part (b) shows an exploded view of the inlet chamber in the anodic compartment, the cathodic outlet chamber is similar, and Fig. 1 part (c) represents the distribution chamber of electrolyte at each compartment. The solid and dashed arrows represent the electrolyte flow at the anodic and cathodic compartments, respectively. Thus, the solution was fed in the inlet chamber of the anodic compartment by means of a perpendicular nozzle. The double convergent channel inside the anodic compartment was achieved by using a gasket of the appropriate shape as it is shown



**Fig. 1.** (a) Schematic representation of the parallel plate electrochemical reactor with convergent flow. (b) Exploded view showing the electrolyte inlet chamber in the anodic compartment. (c) Exploded view showing the final electrolyte distribution chamber at each compartment. (1) plates; (2) anode; (3) current feeders to the anode; (4) cathode; (5) current feeders to the cathode; (6) separator; (7) connexion to Luggin capillaries; (8) gaskets; (9) expanded plastic meshes; (10) electrolyte inlet; (11) electrolyte outlet; (12) electrolyte connexion between anodic and cathodic compartments through a hole in the separator; (13) electrolyte distribution chambers. Solid arrows: electrolyte flow in the anodic compartment. Dashed arrows: electrolyte flow in the cathodic compartment.

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