

VOLTAMETRIC METHOD FOR THE DETERMINATION OF SULPHUR: A PLASTICIZED SULPHUR CONSTRUCTION MATERIAL

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A new voltametric method for the determination of sulphur has been developed. The method, though general, was particularly found useful in the monitoring of plasticization reaction of sulphur where many analytical techniques were found inapplicable.

INTRODUCTION

Sulphur has found many uses but one of the most interesting one and pertinent in the present case has been its use in low-cost housing as sulphur concrete, mortar, bricks, and possible use as drain pipe, flooring materials, decorative tiles and corrosion and seepage resistant materials [1]. A review of these works can be found in literature [2].

The above mentioned uses of sulphur are based upon the plasticization of sulphur by plasticizers like anthracene, dicylopentadiene, thiokol etc. Sulphur with filler and some plasticizer (1% to 5% w/w) is heated to 140^o for a few hours depending upon the nature of the filler, the plasticizer and the products desired. It is always desirable to monitor these reactions and to determine the sulphur content in these products.

During the course of the present investigations of plasticization of sulphur [3], various techniques were employed to monitor the reactions and hence to monitor the sulphur content of the product. These techniques included spectroscopy (UV-Vis, IR: NMR), viscometry, refractometry, polarography (fractional), vacuum sublimation and solvent extraction methods.

Sulphur has always been found to be somewhat difficult to determine quantitatively, though techniques have been developed to determine sulphur in reaction products or as impurity element in other materials [4]. The method which was found to be the most suitable in the present studies being the polarographic technique (at dropping mercury electrode and stationary electrode). It seems that the present method is quite general and suitable for the determination of sulphur in various products. The method and the results are presented below.

A Heath Polarographic Assembly EU 402 in conjunction

with a Beckman 10" linear chart recorder was used. For polarography capillary of 4-6 sec. drop time was used as dropping mercury electrode, whereas for linear scan voltametry a hanging mercury drop electrode was used as stationary working electrode. A standard calomel electrode was used as reference electrode. Some of the experimental details have been described before [5].

PROCEDURE

Pure sulphur was used for the purpose of calibration. Solutions of concentrations ranging from $1 \times 10^{-3}M$ to $1 \times 10^{-2}M$ of sulphur in dimethyl formamide were prepared. Lithium chloride was used as supporting electrolyte. The system was deaerated with nitrogen. Sweep rates of 0.1 V/min. and 2 V/min. for polarography and linear scan voltammetry respectively, were used. A calibration curve between diffusion current (or peak current) and concentration was constructed. 0.0013 g of plasticized reaction product (at various intervals of time) was then dissolved in 20 ml of DMF containing 0.1M LiCl, and the voltammograms recorded.

Table 1. Diffusion and peak currents for calibration

S.No.	Conc. (molar)	Diffusion current (microamp.)	Peak current (microamp.)
1.	1×10^{-2}	142.0	150.0
2.	8×10^{-3}	114.0	130.0
3.	6×10^{-3}	86.0	115.0
4.	4×10^{-3}	54.0	87.0
5.	1×10^{-3}	13.5	30.0

RESULTS

In Tables 1 and 2 the polarographic and voltametric data are collected for pure sulphur (Table 1) and of the reaction products at various interval of time (Table 2). The experimental polarograms and voltamograms etc. are also shown in Fig. 1-4. These voltamograms clearly show the dependence of the diffusion/peak current on the con-

Table 2. Monitoring of sulphur during plasticization reaction

S.No.	Reaction time (minutes)	Limiting current (microamp.)	Peak current (microamp.)
1.	0	146	168
2.	60	140	160
3.	120	138	156
4.	420	132	148
5.	600	122	143
6.	24 hrs.	116	130

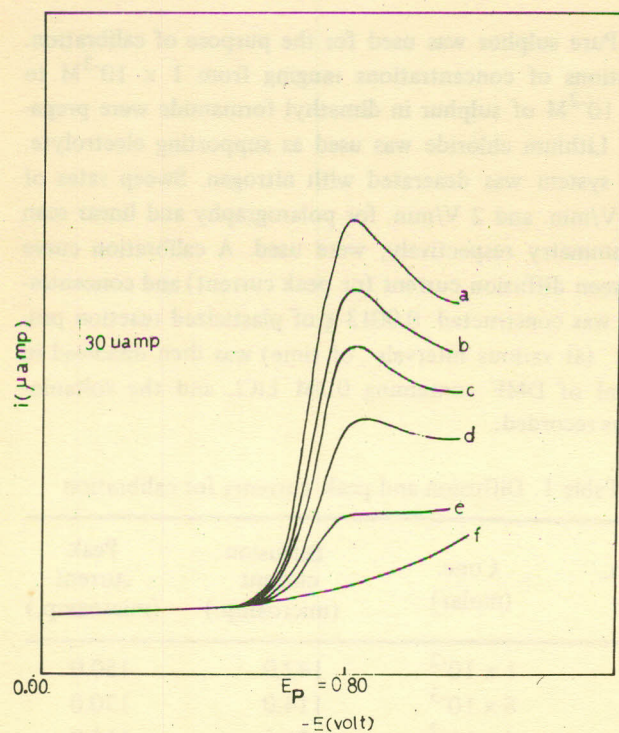


Fig. 1. Linear scan voltamograms of sulphur for calibration; concentration (M), (a) 1×10^{-2} , (b) 6×10^{-3} , (c) 4×10^{-3} , (d) 2×10^{-3} , (e) 1×10^{-3} and (f) base line

centration of sulphur. From the calibration curve the concentration of unreacted sulphur could be read directly.

It must be mentioned here that the recommended method for the determination of sulphur, though found to work for sulphur in other systems [6], was not found applicable in the present investigations (however, see ref. 7). Thus the proposed method seems more versatile and useful.

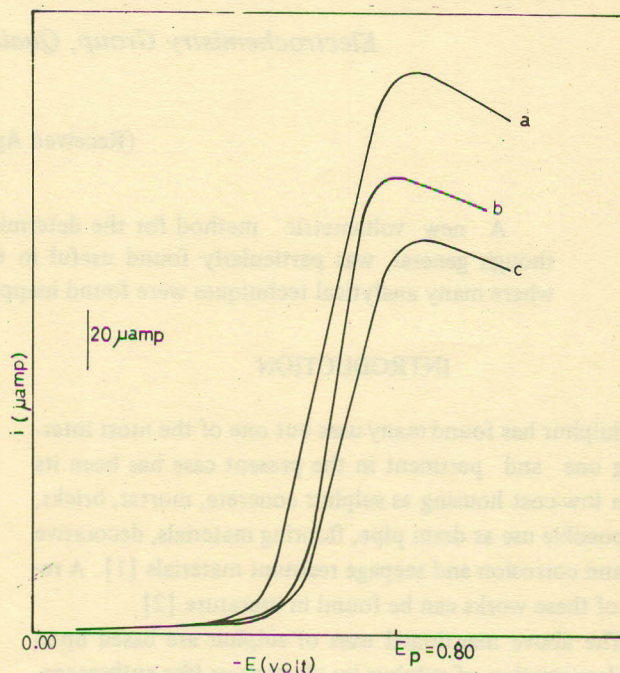


Fig. 2. Sulphur in reaction products (a) $t = 0$, (b) $t = 60$ min. and (c) $t = 120$ min.

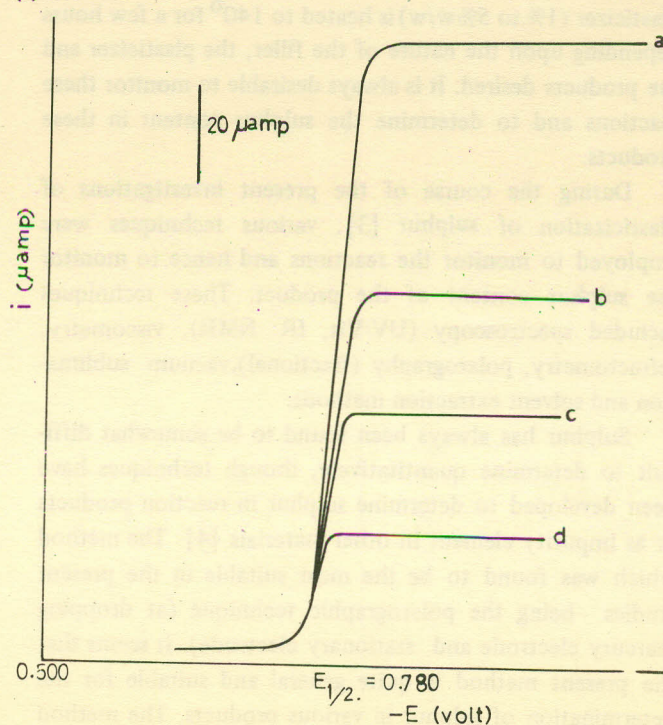


Fig. 3. Polarogram profiles of sulphur for calibration; concentration (M), (a) 10^{-2} , (b) 6×10^{-3} , (c) 4×10^{-3} , and (d) 2×10^{-3} .

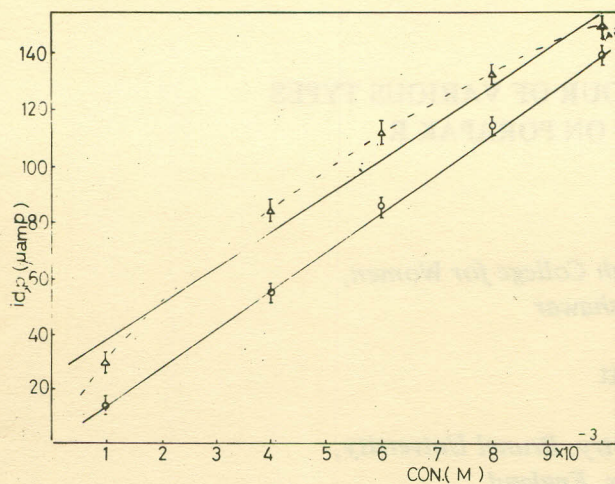


Fig. 4. Plots of i_p vs conc. (Δ), and i_d vs cons. (O).

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