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# SULPHUR DYES: A CONTRIBUTION TO A NEW APPROACH ON THE QUANTITATIVE DETERMINATION

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**Abstract:**An important problem with sulphur dyes application concerns his quantitative determination during dyeing process. Spectrophotometry methods are the most applied ones, although not always the most suitable for this kind of products. In this paper, we will present an approach to a new way to sulphur dye concentration control, based in a potentiometric determination.

**Keywords:**Sulphur Dyes, Dyeing Process, Quantitative Determination

## 1.Introduction

Cotton concerns, in our days, almost half of the world's fiber consumption and this position is predictably stable [1] . Several kinds of dyes can be used in cellulosic fibres, such as direct, azoic, reactive, vat and sulphur dyes. Recent problems with the azo compounds, which are either known or suspected to be allergic, poisonous or carcinogenic, led to a German Regulation in July 1994 that bans some of the most popular dyes [2,3] .

This situation has been a special opportunity for the development of other dye classes, including traditional ones, as sulphur dyes. These are compounds with a long list of critical points, specially in which concerns free sulphides, old application systems, auxiliaries and oxidized dye in the baths and difficult effluent treatment. However, with the recent so-called "ecological" dyes (some of them are above eco-labels request like Oko-Tex Standard 100) and the fact that sulphur dyes are free from heavy metals and have no AOX forming constituents [4] , a stabilised or even a larger world consumption of these products are predictable [5]. Actually, the modern sulphur dyes are, in many cases, the most suitable choice for cellulosic fibres, concerning economical or ecological terms.

A major problem with sulphur dyes is the quantitative determination during the dyeing process or, in other words, the dye concentration control in the bath. Usually, determination methods concerning spectrophotometry are the most applied ones. These methods are, however, not always the most suitable, considering all the problems that can arise from the fact that sulphur dyes are water insoluble products when in the oxidized form. In this paper, we will present an approach to a new way to sulphur dye concentration control in dye baths. The proposed method is based on a potentiometric determination.

## 2.Materials and Methods

This study concerns a traditional sulphur dye (C.I.Sulphur Black 1) and another one in a pre-reduced form (C.I.Leuco Sulphur Black 1). The proposed method is a potentiometric titration, based on sulfide determination by potassium ferricyanide [6].

## 2.1.Equipment

-Crison 2000 potentiometer
-Ingold Pt combined electrode, (Ag/AgCl/3M KCl)
-Glass measurement cell
-Magnetic stirrer

## 2.2.Experimental procedure

After complete oxidation of a 20 ml sample of dye solution (or dyeing bath) with potassium ferricyanide solution 0.1N, the remaining salt is potentiometrically titrated with sodium sulfide 0.1N.
A dye concentration range of 0-6 $gL^{-1}$ was considered. The dyeing baths were prepared according suppliers indications. The experimental conditions consisted on room temperature, closed vessel and stirred solution.

## 3.Results

Experimental results are presented on figures 1 and 2.

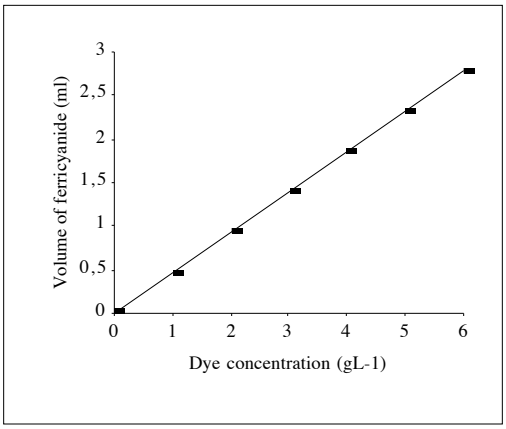


**Figure 1**-Aqueous solutions of C.I.Leuco Sulphur Black 1

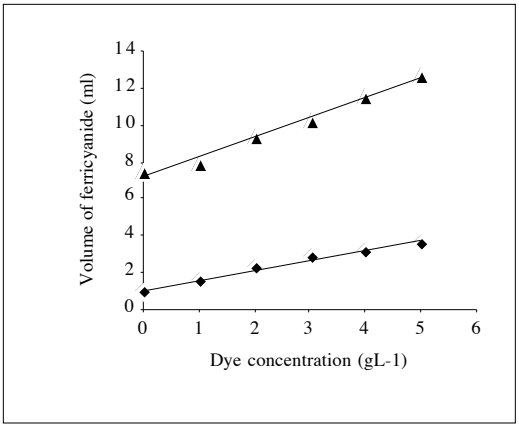


**Figure 2**-Simulated dye baths of C.I. Sulphur Black 1 ▲ and C.I.Leuco Sulphur Black 1 ◆

Figure 1 presents results concerning aqueous solutions of the dye in a pre-reduced commercial form. In figure 2, results concernig simulated dye baths are presented. A traditional commercial dye form and a pre-reduced one are compared.
In both cases, we can observe a linear variation between dye concentration and volume of ferricyanide necessary to his complete oxidation. In simulated baths situation we can observe that a greater volume of oxidant is necessary, probably caused by auxiliary products interference.

## 4.Conclusions

Considering presented results we can preview the possibility of a quantitative determination by a potentiometric method, in aqueous solutions and dyeing baths of traditional and pre-reduced sulphur dyes.

The presence of auxiliary products can be particularly important in which concerns total volume of oxidant. Therefore, dyeing baths composition must always be considered.

Finally, such a method could be a better way of dye concentration control, in order to reduce pollution related to this kind of products.

## 5.References

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