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PRECONCENTRATION OF SUDAN I DYE USING B-CYCLODEXTRIN BUTANEDIOL DIGLYCIDYL ETHER POLYMER AS THE SOLID PHASE EXTRACTANT

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ABSTRACT

In this study, a solid phase extraction method has been developed for the preconcentration of Sudan I at trace level using β-cyclodextrin polymer. After preconcentration, the dye solute was determined by UV-VIS spectrophotometry. Various parameters, such as effect of pH, sample solution, shaking time, amount of adsorbent, agitation time for the % uptake of Sudan I has been optimized. This method has been applied for the determination of Sudan I in different food samples.

KEYWORDS: Sudan I dye, β-Cyclodextrin epichlorohydrin polymer (β-CDP), Preconcentration, Spectrophotometry.

INTRODUCTION

For food industries, color is the first notable characteristic of a food. Colorants are often added to food to enhance its visual aesthetics and promote sales. Color additives reinforce the colors already present in the food and ensure its uniformity. Sudan I is a synthetic azo dye, and is used in many fields such as paint or textile colorants, and floor or shoe polishes. Sudan I can cause tumors in the liver and bladder of mice, and is a possible human carcinogen and mutagen[1], therefore it has been classified as the third category carcinogen by the International Agency for Research on Cancer (IARC). As a result, Sudan I is forbidden as an additive of foodstuffs in any national and international food regulation act. Recently in some places this harmful dye was used as an additive of foodstuffs illicitly to boost up the typical reddish color of chilli powder for the commercial benefits. Therefore, it is required to develop accurate and fast methods to identify Sudan I in foodstuffs. Several methods has been proposed to detect dyes in food stuffs, such as liquid chromatography-photodiode array detection [2,3], molecularly imprinted solid phase extraction [4] and plasmon resonance light

scattering [5]. Common methodologies include HPLC with ultra violet detection [6], enzyme-linked immunosorbent assay [7], atmospheric pressure chemical ionization-tandem mass spectrometry [8], reversed- phase liquid chromatography-electrospray-tandem mass spectrometry [9]. Some methods are very costly are required the expert hands. So, spectrophotometry is widely used for the determination of dyes due to its higher sensitivity, low cost, low interference level and its excellent detection limits. So, Sudan I dye has been determined by spectrophotometric methods after preconcentration using β-CDP in food samples.

Supramolecular complexes with β-cyclodextrin has been a very active research field in past few years [10,11] β-cyclodextrin (β-CD) is very stable oligosaccharide that is composed of seven glucose units linked with other by α-(1,4)-glycosidic linkage. It can form superamolecular complexes with several organic compounds by incorporating them into their hydrophobic cavities. Two or more β-cyclodextrin covalently linked with each other are the polymers. These β-cyclodextrin polymer have been used for the preconcentration of various analytes [12-15]. In the present work, β-cyclodextrin epichlorohydrin polymer (β-CDP) has been used as a solid support for the preconcentration of Sudan I.

Experimental

Reagents and equipments

Apparatus

A Shimadzu UV-1800 spectrophotometer (Shimadzu Ltd., Japan) equipped with the matched 10-mm quartz cells was used to measure absorbance. Digital century pH-meter C_p-901 with a combined glass electrode was used to carry out pH measurements. A thermostatic shaking water bath (Perfit India Ltd.) was used to carry out all the inclusive procedures.

Reagents

All chemicals used were of Anal R grade unless otherwise stated. Double distilled water was used throughout the experiment. Sudan I dye solution was prepared by dissolving 0.248 g in 100ml of double distilled water to give 0.01M standard stock solution and further diluted as and when required. 20g of β-CD was dissolved in 50ml of 20% NaOH. To this was added 20ml of butanedioldiglcidyl ether drop wise. The polymer was formed in 1.5h and dried at 90ºC in oven. The polymer was washed with double distilled water 5-6 times. Then, the polymer was dried again at 90ºC and kept at room temperature in desiccator for further use.

Buffer solution in the pH range of 2.0-3.5 were made by mixing equimolar solutions of hydrochloric acid/Sodium Acetate and buffer solutions in the pH range of 4.0-6.5 were made by mixing equimolar solutions of sodium acetate and acetic acid solutions in the different proportions While those in the pH range of 7.0-11.0 were made by mixing equimolar solutions of ammonia and ammonium chloride. The glass wares were washed with chromic acid and soaked in 5% nitric acid and then cleaned with double distilled water before use and dried in an electric oven.

Procedure

300mg of β-CDP and 2.5 mL of buffer solution (pH 3.0) were added to a 100 ml stoppered conical flask at room temperature. The mixture was allowed to stand for 5 min. 3ml of dye was added and made up to 75 ml with double distilled water. After the mixture was shaken in the thermostatic shaking water bath for 90min., 5.0ml of supernatant solution was transferred into a 10ml volumetric flask and the absorbance was measured using spectrophotometric method.

RESULTS AND DISCUSSION

Optimization of various parameter

Effect of pH

The complexation of the dye with the polymer is dependent on the pH of sample solution. The pH of the solution was adjusted in the range of 1 to 4 using different buffer solutions and then the preconcentration procedure as described was applied. As it can be seen in "Fig. 1", quantitative uptake (> 95%) was obtained at 2.0. Therefore, the working pH was chosen as 2.0 for the following experiments.

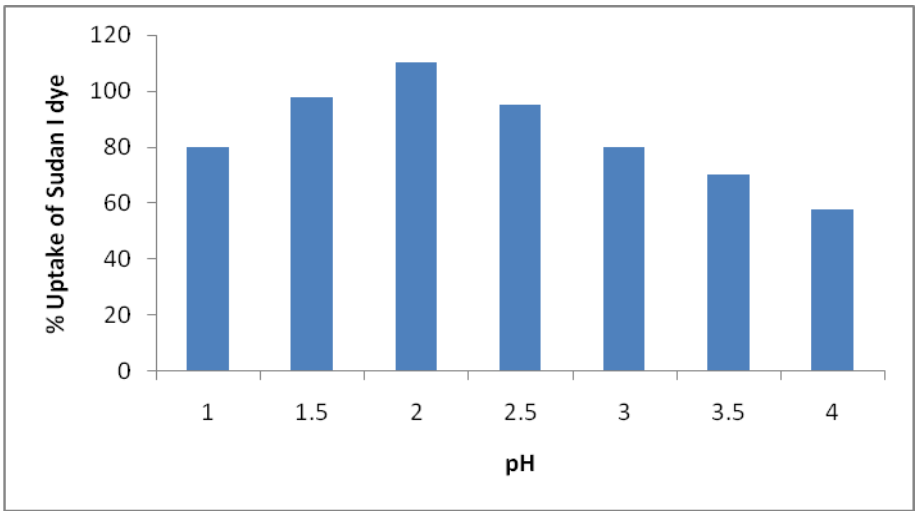

Fig. 1. Effect of the pH on the % uptake of the Sudan I dye by the polymer

Effect of the shaking time

The Shaking time is an important factor in determinating the possibility of application of the B- CDBP polymer for the selective % uptake of the metal ion. Different shaking time (ranging from 15 to 160 min.) were studied for the % uptake of Sudan I by β-CDBP polymer. The results of the % uptake of Sudan I vs. the shaking time "Fig. 2" show that the % uptake reach maximum above (> 95 %) at 160 min. Therefore, the shaking time of 120 min. was selected as the adsorption equilibrium time.

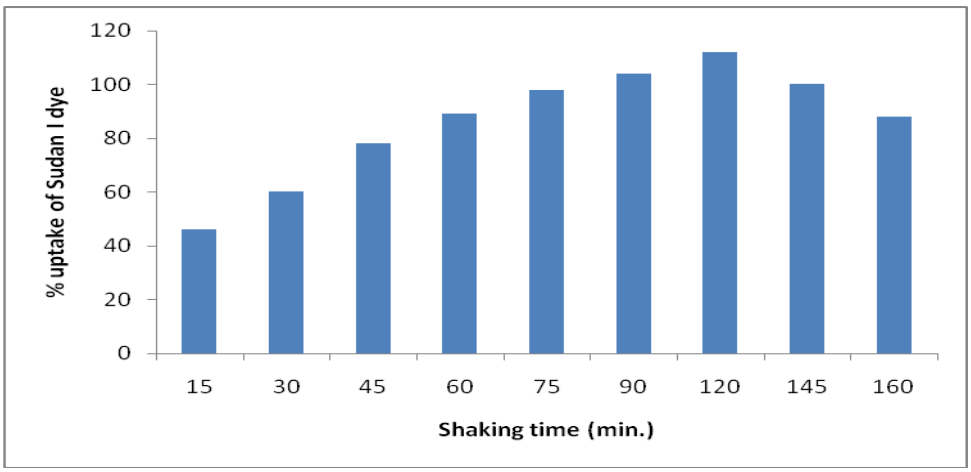

Fig. 2.Effect of shaking time on the % uptake of the Sudan I by the polymer

Effect of sample volume

In order to explore the possibility of enriching low concentration of analytes from large volume of solution, the effect of sample volume on the retention of Sudan I was also investigated. For this purpose, 15, 30, 45, 60, 75, 90, 105 mL of sample solutions were taken. Quantitative uptake (> 95 %) was obtained for the sample volume of 90 mL "Fig. 3". Therefore, 90mL of the sample solution was adopted for the preconcentration of analyte from sample solutions.

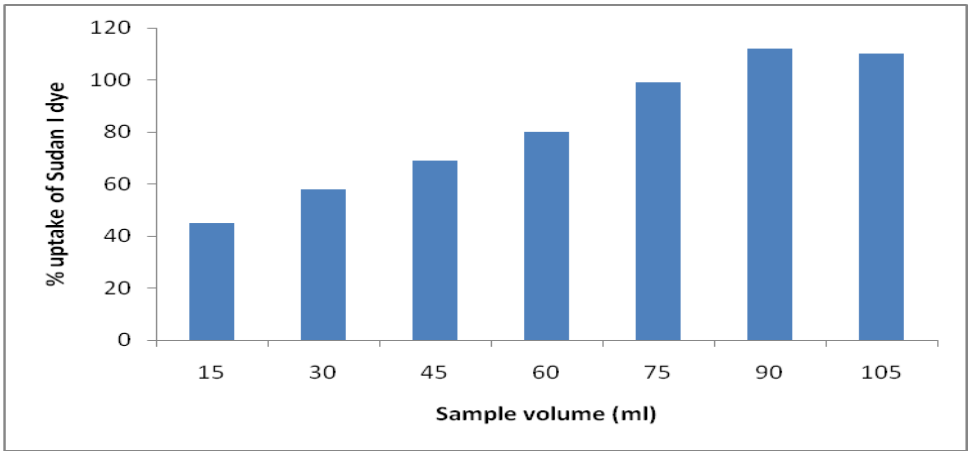

Fig. 3. Effect of sample volume on the % uptake of the Sudan I by the polymer

Effect of amount of polymer

The amount of the β-CD polymer is another important parameter that affects %uptake of dye. A quantitative removal (≥95%) cannot be achieved when the β-CD polymer is less than the optimum amount. In order to optimize the smallest amount of polymer, 100 mg, 200 mg, 300 mg, 400 mg and 500 mg of the polymer were added to the solution containing known amount of dye. The quantitative recoveries were obtained at 300 mg of β-CD shown in "Fig. 4". Therefore, 300 mg of the β-CD has been used for further studies.

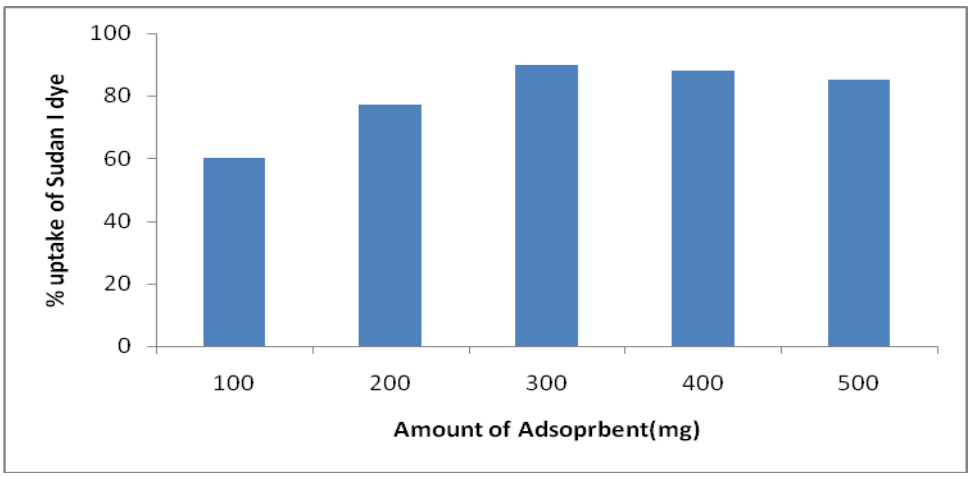

Fig. 4.Effect of amount of adsorbent on the % uptake of the Sudan I by the polymer

Effect of agitation speed

Speed of shaking is the important factor in determining the possibility of application of polymer for the quantitative %uptake of Sudan I dye. Different speed (ranging from 40 to 140 r.p.m) were studied for the % uptake of Sudan I dye by polymer. The results of % uptake of Sudan I vs. agitation speed "Fig. 5" shows that the % uptake reach maximum (≥95%) at 120 r.p.m. Therefore, the shaking speed of 120 r.p.m. was selected for the further studies.

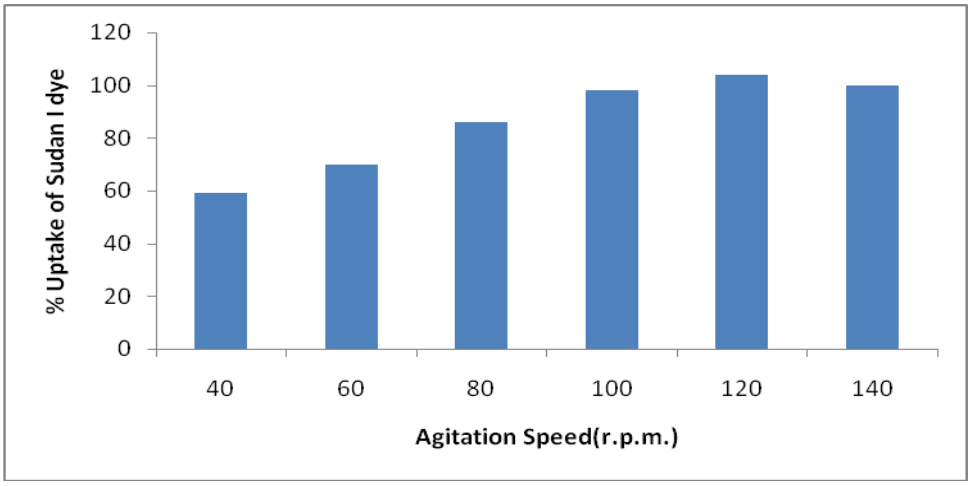

Fig. 5. Effect of agitation speed on the % uptake of the Sudan I by the polymer.

Applications

Determination of samples

The proposed method has been applied for the determination of Sudan I dye in Chilli Powder and Chilli Sauce. The results are given in table.

Table 1: Result of determination of Sudan I in food samples

Food Samples	Added, ug/mL	Found, ug/mL	Recovery, %
[a]Chilli powder	0	0.008	-
	0.275 ug/mL	0.277 ug/mL	100%
	0.551 ug/mL	0.537 ug/mL	97.45%
[b]Chilli Sauce	0	0.015	-
	0.275 ug/mL	0.264%	96%
	0.551 ug/mL	0.550 ug/mL	99.81 %

[a]*Chilli powder - locally available in market,* [b]*Chilli Sauce - locally available in market*

CONCLUSION

The proposed preconcentration method consist of a simple and low cost procedure which permits the quantitative recovery of Sudan I dye from food samples. The synthesis of the polymer is easy and the method has a good accuracy, sensitivity and repeatability. The polymer has been used in all the experiments performed for the study. It has a unique stability and reuseability. This method is convenient for the determination of Sudan I dye in the different food samples like Chilli powder and Chilli sauce.

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