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Preconcentration of Sudan III dye using β-Cyclodextrin Butanediol Diglycidyl Ether Polymer as the Solid Phase Extractant

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Abstract: *A solid phase extraction method for the preconcentration of Sudan III dye at trace level using β-cyclodextrin polymer. The residual concentration of the dye solute was determined by spectrophotometric method. The factors affecting the preconcentration of Sudan III such as pH, shaking time, sample volume etc. were observed and discussed. The recovery of Sudan III dye was found to be ≥ 95%. The proposed method has been applied for the determination of Sudan III in different food samples.*

Keywords: *β-cyclodextrin butanediol diglycidyl ether polymer (β-CDP), Sudan III dye, preconcentration, Spectrophotomertry.*

1. INTRODUCTION

Synthetic dyes are widely used for improving the color and visual aesthetic appeal of some food and this effect is maintained throughout the production process and during storage. They present high stability to light, oxygen and pH changes and have lower prices compared to natural dyes[1,2]. Sudan III is synthetic industrial azo-dye, traditionally used in waxes, plastics, oils, and polishes. The dye is categorized as class 3 carcinogen by International Agency for Research on Cancer (IARC). Although recognized as carcinogen, Sudan III have been found recently in food products in some European countries. Sudan dyes are added to food such as chilli powder to mimic, intensify, and prolong the appearance of natural red hues. In UK, more than six hundred products containing Sudan dyes have been recalled such as fish sauce, Worchester sauce, noodle soup, and pizza. Therefore, they are illegal to use as food additives, according to the FAD and EU. The European Commission requires products to have documentation confirming the absence of Sudan dyes. The EU has set the detection limit at 0.5-1 mg/kg for Sudan dyes, and any food material containing more those limits should be withdrawn the market. The analytic techniques frequently employed for the determination of the colors include thin layer chromatography[3], capillary electrophoresis[4], HPLC coupled with photodiode array[5], chemiluminescence[6], electrochemical with carbon nanotube modified electrodes[7] and inter-laboratory comparison[8]. In all instances, most of these methods require a highly qualified operator and high cost instrumentation. Thus, a highly sensitive and low cost method is still needed for the development in the field of analytical chemistry. So, Sudan III dye has been determinated by spectrophotometric methods after preconcentation using β-CDP in food samples.

Supramolecular complexes with β-cyclodextrin has been a very research field in past few years[9,10] β-cyclodextrin (β-CD) is a very stable oligosaccharide that is composed of seven glucose units linked with each other by α-(1,4)-glycosidic linkage. It can form supramoleculer complexes with several organic compounds by incorporating them into their hydrophobic cavities. Two or more β-cyclodextrin covalently linked with each other are known as polymers. These β-cyclodextrin polymer have been used for the preconcentration pf various analytes[11-14]. In present work, β-cyclodextrin 1,4-butanediol diglycidyl ether polymer (β-CDP) has been used as a solid support for the preconcentration of Sudan III dye.

2. 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 the chemicals used were of Anal R grade unless otherwise stated. Double distilled water was used throughout the experiment. Sudan III dye solution was prepared by dissolving 0.352 g in 100ml of double distilled water to give 0.01M standard stock solution and further dilutions were made as 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

200mg 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.

3. RESULTS AND DISCUSSION

Optimization of Various Parameters

Effect of pH

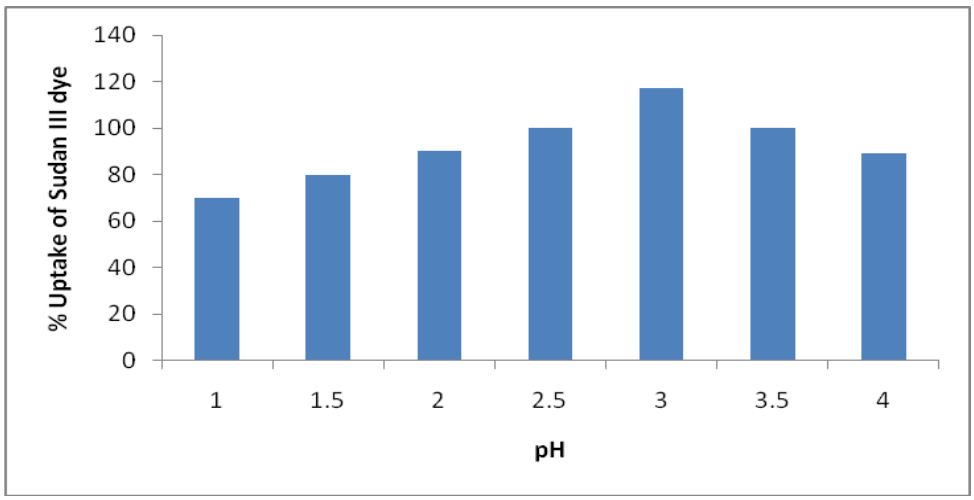

Figure 1. *Effect of the pH on the % uptake of the Sudan III dye by the polymer*

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 3.0. Therefore, the working pH was chosen as 3.0 for the following experiments.

Effect of the shaking time

The Shaking time is an important factor in determinating the possibility of application of the β-CDBP polymer for the selective % uptake of the Sudan III dye. Different shaking time (ranging from 15 to 120 min.) were studied for the % uptake of Sudan III by β-CDBP polymer. The results of the % uptake of Sudan III vs. the shaking time (fig. 2) show that the % uptake reach maximum above (≥ 95 %) at 90 min. Therefore, the shaking time of 90 min. was selected as the adsorption equilibrium time.

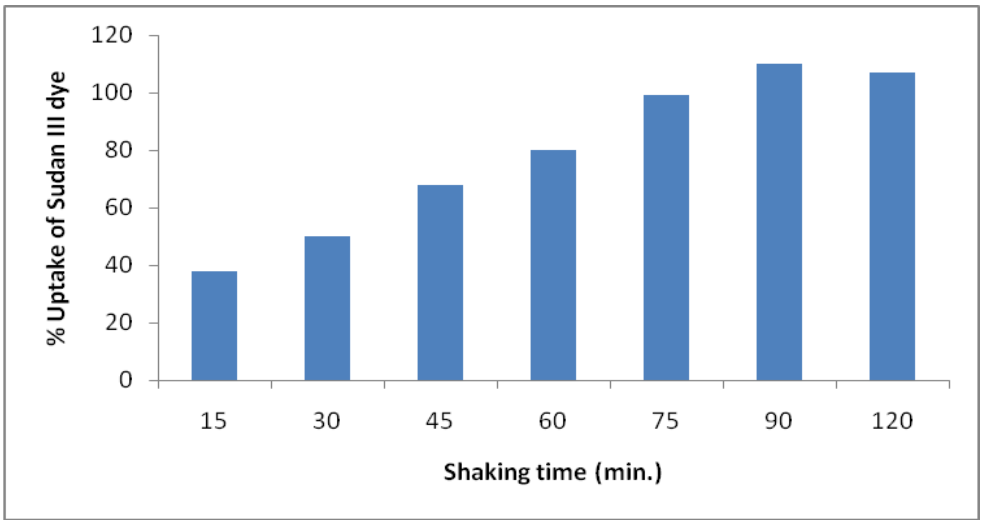

Figure 2. *Effect of shaking time on the % uptake of the Sudan III by the polymer*

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 75 mL (fig. 3). Therefore, 75mL of the sample solution was adopted for the preconcentration of analyte from sample solutions.

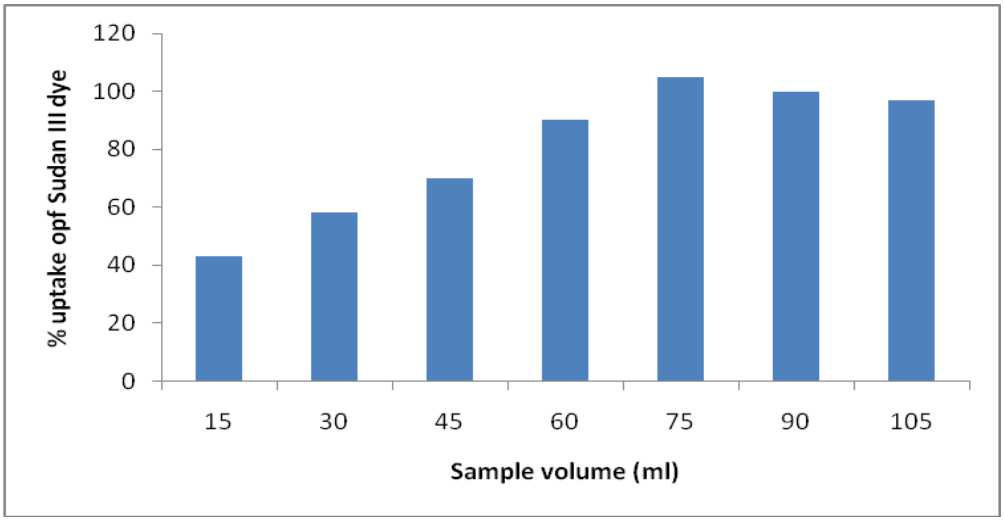

Figure 3. *Effect of sample volume on the % uptake of the Sudan III by the polyme*

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 200 mg of β-CD shown in (Figure 4). Therefore, 200 mg of the β-CD has been used for further studies.

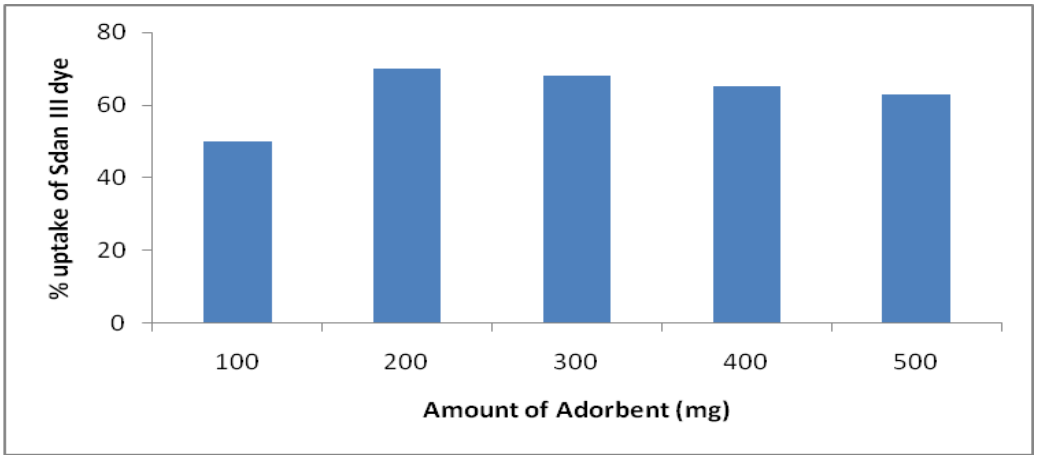

Figure 4. *Effect of amount of adsorbent on the % uptake of the Sudan III by the polymer*

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 III dye. Different speed (ranging from 40 to 140 r.p.m) were studied for the % uptake of Sudan III dye by polymer. The results of % uptake of Sudan III vs. agitation speed (Figure 4) shows that the % uptake reach maximum (≥95%) at 80 r.p.m. Therefore, the shaking speed of 80 r.p.m. was selected for the further studies.

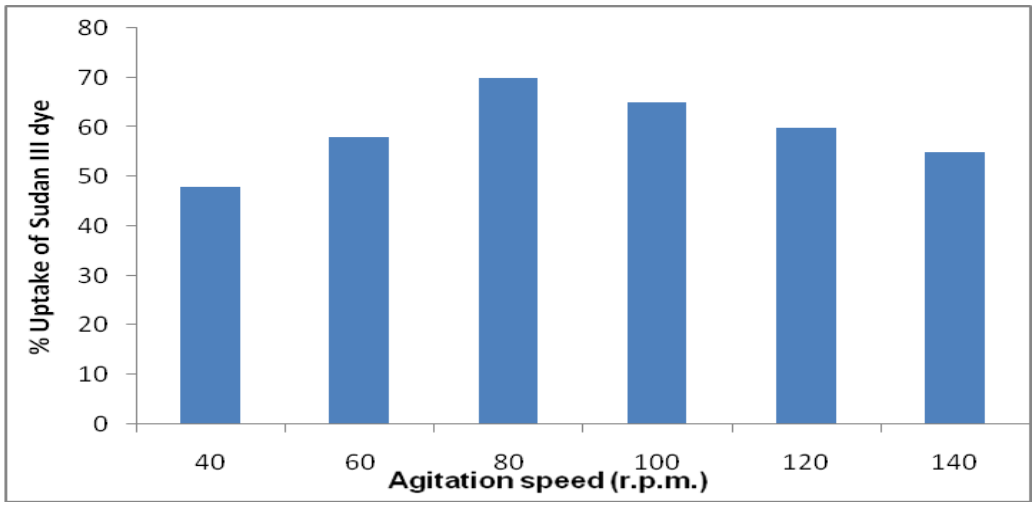

Figure 5. *Effect of agitation speed on the % uptake of the Sudan III by the polymer.*

4. APPLICATIONS

Determination of samples

The proposed method has been applied for the determination of Sudan III dye in Curry powder and Navratan oil. The results are given in table.

Table 1. *Result of determination of Sudan III in food samples*

Food Samples	Added,ug/mL	Found,ug/mL	Recovery, %
[a]Curry powder	0	0.010	-
	0.440 ug/mL	0.405 ug/mL	92.04 %
	0.920 ug/mL	0.898 ug/mL	97.60 %
[b]Navratan oil	0	.019	-
	0.469 ug/mL	0.439 ug/mL	93.60 %
	0.939 ug/mL	0.939 ug/mL	100 %

[a]Curry powder – locally available in market, [b]Navratan oil – locally available in market

5. CONCLUSION

The proposed preconcentration method consist of a simple and low cost procedure which permits the quantitative recovery of Sudan III 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 III dye.

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