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# Design of Kinetic Spectrophotometric Method for Sensing Tartrazine Color in Real Samples Using Sensor Starch-capped ZnSNPs

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**Abstract**

Tartrazine is a synthetic organic food dye that can be found in common food products such as bakery products, dairy products, candies, and beverage. The presence and content of tartrazine color must be controlled in food products due to their potential harmfulness to human beings. To determine the tartrazine color in solution, we used a prepared from Starch-capped ZnSNPs sensor and kinetic spectrophotometric method. The calibration curve was linear in the range of 0.01 to 10.0 mg $L^{-1}$. The standard deviation of 1.0 %, and detection limit of the method (0.01 mg $L^{-1}$ in time 25 min, 399 nm) were obtained for sensor level response Starch-capped ZnSNPs with (95%) confidence evaluated. The chemical Starch-capped ZnSNPs sensor made it possible as an excellent sensor with reproducibility.

**Keywords:** Color Tartrazine, Determination, Real samples, Sensor, Starch-capped ZnSNPs.

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## Introduction

Wastewater of textile, paper, rubber, plastic, leather, cosmetic, food, and drug industries contains dye and pigments which are hazardous and can cause skin irritation, cancer, and mutation in living organisms. Also, it can be the cause of eye burns and conjunctivitis in both human, and animals. Also, inhalation of them can affect the respiratory tract with symptoms of rapid or difficult breathing while mouth ingestion can affect gastrointestinal tract with symptoms of burning sensation, nausea, vomiting, hyperhidrosis disorder, cognitive impairment disorder, micturition disorder, and methemoglobinemia–like syndromes [1].

Due to the toxic nature of some dyes, their mutagenic impacts, and skin disorders (irritation and allergies), the potential pollution dyes, and their intermediates have drawn the attention of many researchers. In addition, microbial degradation of synthetic dyes to carcinogenic impact of benzidine and other aromatic compounds has been a matter of health concern [2]. Food dyes are often added to foodstuffs and drinks in order to supply, intensify or restore their color advantages compared with natural dyes such as high stability to light, oxygen and pH, color uniformity, low microbiological contamination and relatively lower production costs.

Tartrazine is a synthetic organic food dye that can be found in common food products such as bakery products, dairy products, candies, and beverages [3] which must be controlled in produced food and content of the TZ color which is potentially harmful to human beings [4]. Therefore, is very important to determine the TZ color in commercial food products.

So far, different methods such as Chromatography [5,6], Chromatographic by HPLC [7], Electro analytical [8,9], colorimetric [10], Oxidation [11], voltammetry [12], flow injection [13], adsorption [14], and spectrophotometry [15,16] have been used for this purpose. Regardless of how time-consuming these techniques are, they need advanced instrumentation to fit real-time analysis.

Accordingly, developing a simple, quick, elective and delicate method like spectrophotometric measurement in determining the TZ color was highlighted. In this method sensing the TZ color was done with high sensitivity and excellent electivity for discerning and accurate reorganization of species (1-inorganic 2- organic and 3- biomolecules) in different intricate matrices. Attention has been drawn to the noble metal nanoparticles-based UV–visible spectrometric methods [17].

Due to the profitable application of metal nanoparticles in technologies, they have taken advantage of the nano-scale materials in a variety of fields from chemistry to medicine [18,19]. Recovery of nanoparticles from the plant tissue is tedious, expensive, and requires enzymes to

destroy plant cellulose tissue. Therefore, the used small molecular polymer substrates in low and large-scale processing to prepare various metal nanoparticles.

In recent years, the use of plant extracts for preparation of metal nanoparticles has been proposed as an easy and suitable alternative to chemical and physical methods [20]. The forms, sizes, and structures of metallic nano materials extensively linked to their chemical, physical, and optical characteristics which set the ground for their successful applications in technologies. This exceptionality arises from the size, form, composition, crystallinity, and structure of ZnSNPs in comparison with its bulk form [21]. The exclusive properties of ZnSNPs have applications in the fields of bio-sensing, and nano medicine, pharmacy, and biomedical engineering of varying sizes and shapes. They have been utilized in a broad range of applications and medical equipment such as electronic devices, paints, coatings, soaps, detergents [22,23].

This study aimed to find a simple, fast, and very sensitive method for identifying and measuring of the TZ color by starch-capped ZnSNPs Sensor. Various factors such as pH, TZ color concentration, starch-capped ZnSNPs concentration, time reaction, ionic strength, etc. are effective in the response of the method and obtaining the optimal test values and the linear range, detection and accuracy of the method presented in the measurement of the TZ color. The method by kinetic Spectrophotometric was introduced to measure TZ color in food samples, which can be used for other samples (Figure 1).

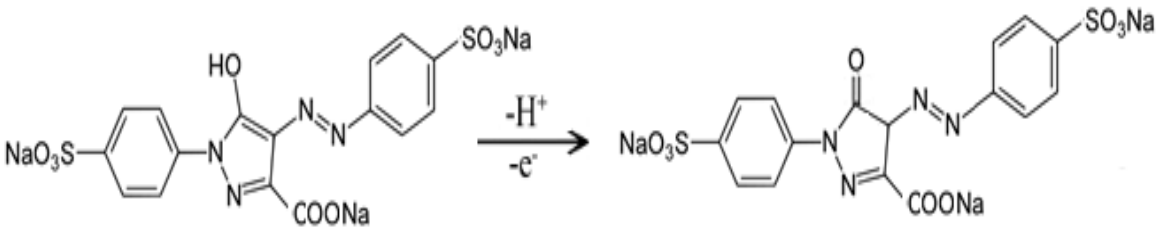


**Figure 1.** Mechanism of the tartrazine (TZ) color on Starch-capped ZnSNPs.

## Experimental

*Reagents and materials*

All chemicals of the lead nitrate $Zn(NO_3)_2$ (assay 99%), sodium sulfide ($Na_2S$) (assay 99.0 %), and Starch from Merck Company, and tartrazine (TZ) color (assay 98.0%) was purchased from India Company. The universal buffer solutions were prepared from 1 ml of boric acid /acetic acid/phosphoric acid (1.0 M). The final pH was adjusted by addition of 0.2M sodium hydroxide

which was bought from Merck Company (Merck, Darmstadt, Germany). DD $H_2O$ (double distilled water) was used in the preparation of the solutions. The ensuing shows the concentrations of the stock solutions.

*Instrumentation*

UV–vis spectrophotometer (Model UV–vis Shimadzu 180, Japan). Fourier transform infrared spectra (FT-IR) were obtained on a (PerkinElmer FT-IR spectrum BX, Germany). Scanning electron microscopy (SEM model KYKY-EM 3200, Hitachi Firm, China) under an acceleration voltage of 26kV) was used to study the morphology of samples. Transmission electron microscopy (TEM, model JEOL, Hitachi Company, China). For the measurement of pH, the pH/Ion meter (model-728, Metrohm Firm, Switzerland, Swiss) was used.

*Pretreatment of real samples*

In a 50 mL beaker, treatment of a 10 mL portion of strawberry jelly (Tehran, Iran), fruity candy (Bon Bon, Iran), Smart beans (Morvarid, Iran), gummy candies (Yupi, Indonesia), Nooshmak (Tehran, Iran), and jell gum with fruit taste (Shiba, Iran). The Pastille samples were dissolved in warm water, filtered and diluted in a volumetric flask. An aliquot of the above sample solutions was treated under the general procedure for ATPS and subsequent determination of TZ color [24].

*Synthesis of Starch-capped ZnSNPs*

The nanoparticle ZnS was synthesized in reactive solution prepared using zinc nitrate $Zn(NO_3)_2$ and sulfide sodium ($Na_2S$) with concentrations of 0.1 M and 0.1 M. The starch pellets were used as a base medium, and its concentration was set to (0.1 M). 20 mL of all the above solutions were prepared separately, using distilled water as a solvent, and mixed together in a beaker. The reactive vessel with solution was immersed into 20 ml acetone maintained at $40^oC$ and pressure of $10^{-5}$ mbar. A thermometer was placed in the vessel to measure the temperature of the bath solution, and also a temperature sensor and a dimer with temperature controller were attached to maintain the constant temperature. The solution was stirred well with the help of magnetic stirrer to maintain the homogeneous mixture. The prepared solution was colorless, and turned yellowish after (30 min) and suddenly changed into gray color which indicated the chemical reactions, and also confirmed the formation of ZnS. The reactive solution was continuously stirred for (2 h). The powder was collected, and dried in a hot air oven [25].

*Procedure kinetic spectrophotometric detection measurements*

The ensuing steps have been considered for a kinetic spectrophotometric method experiment in the current study, at the initial step. Some of the sample solution containing 1 ml of TZ color (10.0 mg $L^{-1}$) was added to a 10 ml volumetric balloon. Then, by increasing the first drop of 1 ml of the starch-capped ZnSNPs solution ($3.0\times10^{-2}$ $molL^{-1}$) into a balloon, the reaction start time is recorded by a timer. After 5 min from the start of the reaction, the solution is stirred for 5 min and an adequate amount of the solution was added to a 1 cm cell subsequently. Finally, the difference between the quantities of the absorption, (399 nm) in a time interval (5.0-25.0 min) was checked by using UV-visible spectrum (AAb).

By adding TZ color to the solution, it was observed that absorbance kinetic spectrophotometric of the Starch-capped ZnSNPs at the wavelength of (399 nm) was dropped. At the same time, with the help of spectrophotometry and UV–visible spectrum (AAb), the apparent spectral evolution including the formation of a well-defined isobestic point at around (399 nm) was estimated. All reaction steps were repeated by increasing the concentration (0.2 mg $L^{-1}$) of the TZ color every 5 min. Moreover, the mentioned steps were repeated for a reaction in the absence of TZ color (Abs b). Eventually, (Abs a) Abs blank – Abs sample was calculated. The reaction of the TZ color by Starch-capped ZnSNPs was detected in the acidic medium in its wavelength (399 nm). Figures 2 and 3 demonstrate the absorption spectra of the TZ color in the solution [20,26,27].

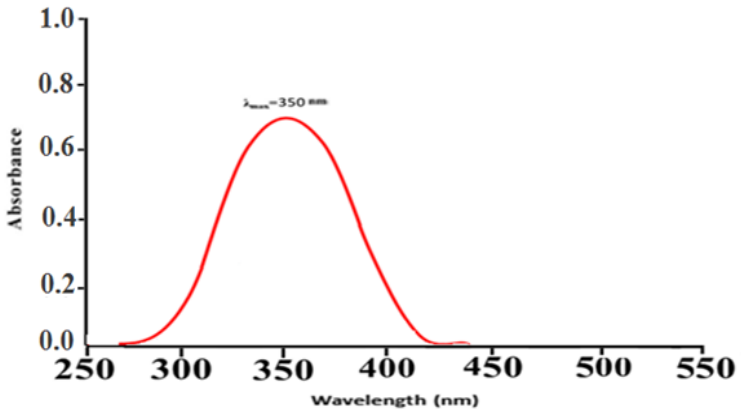


**Figure 2.** The sorption spectra reaction of Starch-capped ZnSNPs

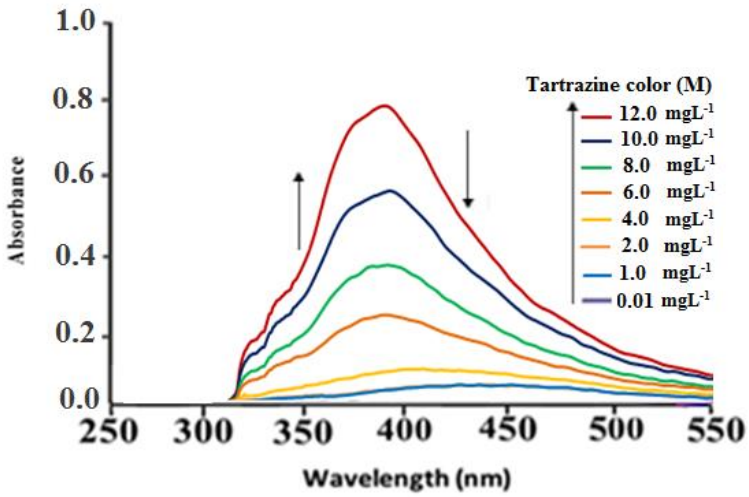


**Figure 3.** The sorption spectra of result Starch-capped ZnSNPs and TZ color 5 min, and increasing concentration of TZ color solution (0.2 mg $L^{-1}$).

## Results and discussion

*Characterization of Starch-capped ZnSNPs Synthesis*

FTIR spectra for Starch-capped ZnSNPs synthesis are shown in Figure 4a. The vibrational frequencies for stretching bonds in ZnS molecule cannot be detected by FTIR analysis. This confirms that ZnS showed no definite absorption peaks in the range 400 - 4000 $cm^{-1}$. The vibration modes located at 3423 $cm^{-1}$ can be assigned to the O–H broad absorption mode due to the hydroxyl group in the compound. The absorption band at 2928 $cm^{-1}$ corresponds to the C-H stretching vibration mode. The broad absorption near 1300 - 1000 $cm^{-1}$ confirms the presence of the C–O bond. The absorption band at 1637 $cm^{-1}$ is due to the O–H bending vibration from the water molecules adsorbed into the surface. Furthermore, no significant difference between the FTIR spectra of Starch-capped ZnSNPs is observed [20,28].

The XRD pattern of the Starch-capped ZnSNPs is shown in (Figure 4b). The synthesized nano powders to be polycrystalline in nature. All detectable peaks corresponding to (111), (420), (331), (400), (222), (311), (220), (200) and (422) planes belong to the pure cubic phase of PbS (JCPDS no. 78–1901) [27]. Figure 4c, 4d are the morphological features, and particle size distribution of the starch-capped ZnSNPs using SEM micrograph. It has been seen that the particles were mostly spherical with a various size distribution as they form agglomerates. We obtained the average particle size distribution within the range of 15-45 nm very close to those determined by XRD analysis [29].

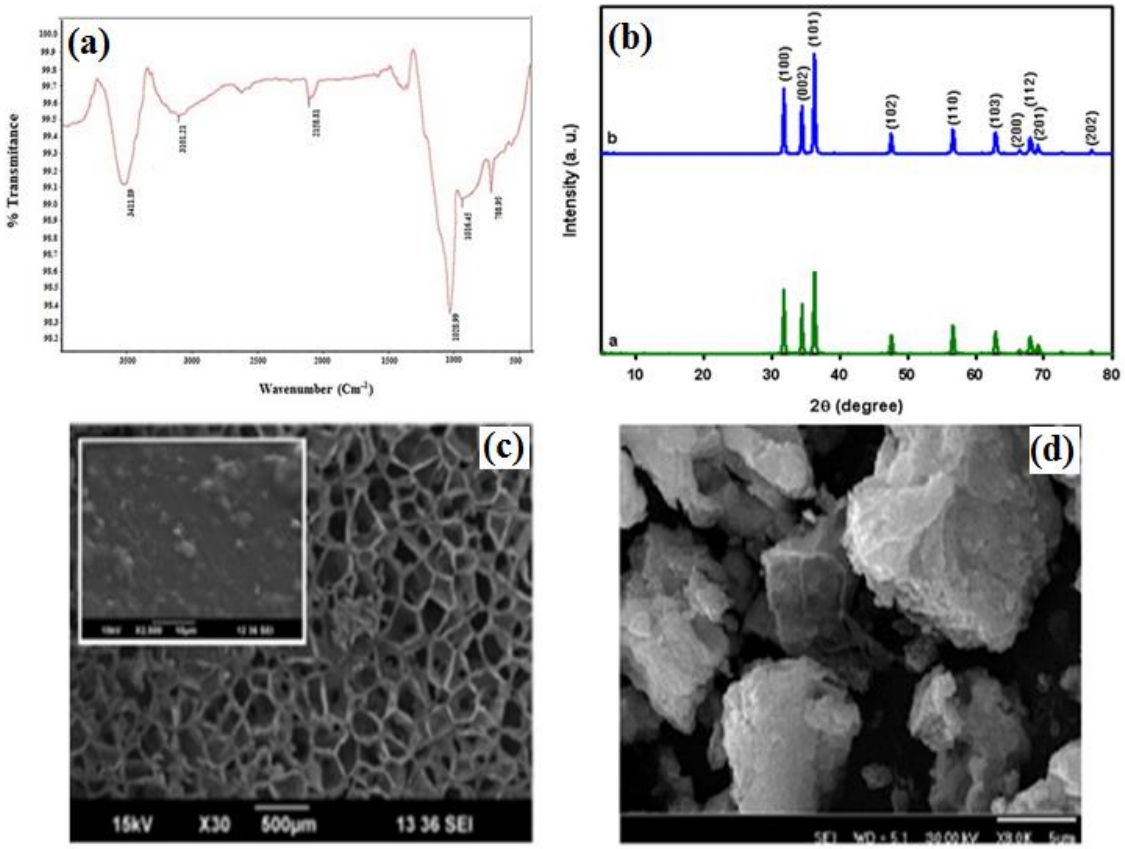


**Figure 4.** (a) FT-IR spectrum image (b) XRD of the preparation of synthesized Starch-capped ZnSNPs (c) SEM image of synthesized Starch-capped ZnSNPs in 500 nm (d) SEM image of synthesized Starch-capped ZnSNPs in 100 nm.

*Optimization of Sensing Conditions*

In order to obtain a highly sensitive response to the detection of the TZ color, the optimization of pH, buffer, starch-capped ZnSNPs, and incubation time were carried out systematically.

*Effect of buffer for rapid detection of TZ color*

The best type of buffers and the volume for maximum absorbance TZ color with Starch-capped ZnSNPs sensor is investigated. To this step, the procedure is as follows: In 10 ml balloon, separately 1 ml of TZ color (10.0 mg $L^{-1}$) and a volume of each type of acetic acid/boric acid/phosphoric acid buffer and then 1 ml of Starch-capped ZnSNPs ($3.0\times10^{-2}$mol $L^{-1}$) was added. Then, after 25 minutes, the adsorption was measured by UV–Visible spectrum. Based on the results, 1 ml of acetic acid as buffer, shows the highest percentage for determining of TZ color, so acetic acid/trichloro acetate buffer (1.0 M) was added to adjust the pH as the optimal buffer [28,29].

*Optimization of pH decomposition*

A very important factor for measurements is the pH of the decomposition solution. To find the best pH for determining the TZ color (10.0 mg $L^{-1}$) a Starch-capped ZnSNPs sensor (pH range from 2 to 10, in 399 nm) performed by the kinetics spectrophotometric method for TZ color was scrutinized (Figure 5) [28,30].

The absorbance difference, in other words, the calculation of the difference between the absorbance of the Starch-capped ZnSNPs and the absorbance of the compound TZ- Starch-capped ZnSNPs at 399 nm would lead to absorbance measurement. Absorbance increased rapidly on changing the pH from 1.0 to 4.0 while it decreased at pH values higher than 4.0. This phenomenon might be because of the weak complexation at lower pH values (pH < 4.0). On the other hand, the reduced response of the proposed Starch-capped ZnSNPs sensor for determining TZ color at pH > 4.0 could be due to a possible formation of the hydroxide of TZ color in solution. Thus, pH 4.0 was selected as a favorable pH for all subsequent experiments [30].

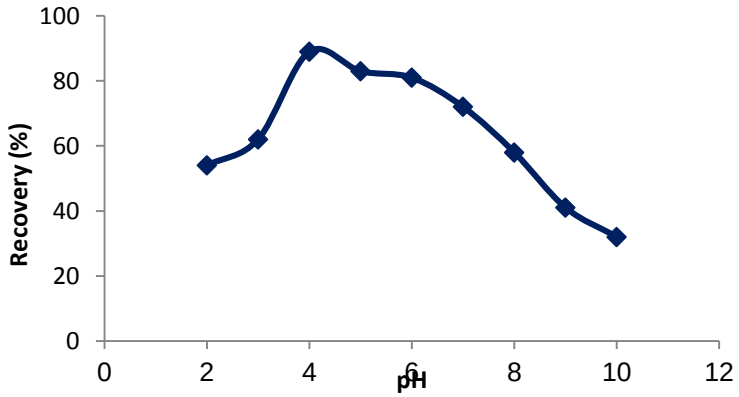


**Figure 5.** The impact of pH on the absorbance, aqueous sample volume, 10 mL in 399 nm. (Starch-capped ZnSNPs, $3.0\times10^{-2}$ M, TZ color = 10.0 mg $L^{-1}$, time 25 min).

*Impact of Starch-capped ZnSNPs on the reaction rate*

To scrutinize the efficacy of Starch-capped ZnSNPs sensor on the reaction rate, 1 ml Starch-capped ZnSNPs ($0.25\times10^{-3}$ to $5.0\times10^{-2}$ mol $L^{-1}$) along with 1 ml TZ color (10.0 mg $L^{-1}$) solution, and buffer acetic acid/acetate (1.0 M) were used to adjust the pH solution, pH=4, in 399 nm). After 25 minutes, the calculation of the difference between the absorbance of the Starch-capped ZnSNPs and the absorbance of the compound TZ-Starch-capped ZnSNPs at (399 nm) was

scrutinized. As demonstrated in Figure 6, and by considering the results, the preferred concentration was selected to be Starch-capped ZnSNPs sensor ($3.0\times10^{-2}$ mol $L^{-1}$) [31].

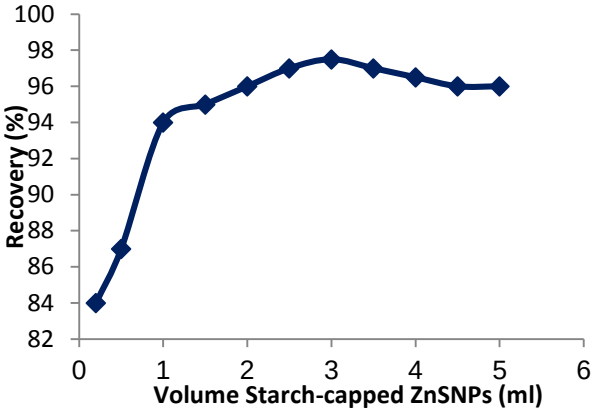


**Figure 6.** The impact of volume Starch-capped ZnSNPs on the absorbance, aqueous sample. 10 mL in 399 nm. (pH =4, TZ color = 10.0 mg $L^{-1}$, time 25 min).

*Impact of time on the reaction rate*

To determine the optimum time of the reaction in a volumetric flask (10 ml), first 1 ml TZ color (10.0 mg $L^{-1}$) solution, 1 ml Starch-capped ZnSNPs ($3.0\times10^{-2}$mol $L^{-1}$), and buffer acetic acid/ acetate (1.0 M) were added to adjust the pH solution. In the 1-10 min interval of time, the sorption of solutions is estimated. In other words, the calculation of the difference between the absorbance of the Starch-capped ZnSNPs, and the absorbance of the compound TZ- Starch-capped ZnSNPs at (399 nm) would lead to absorbance measurement. In Figure 7, alterations of the sorption based on the time at 25°C degrees of temperature are exhibited. The optimum time was selected to be (25 min) [32].

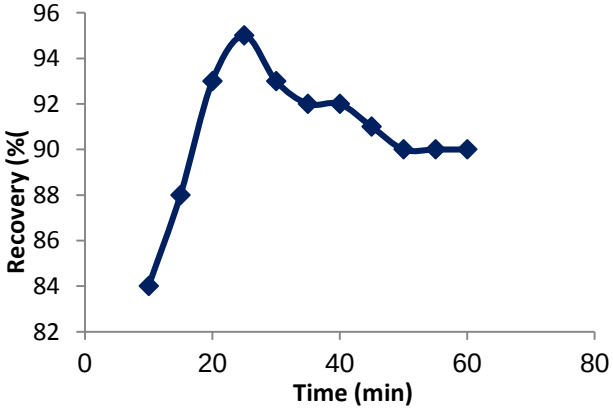


**Figure. 7.** The impact of time on the absorbance, aqueous sample volume, 10 mL in 399 nm. (Starch-capped ZnSNPs, $3.0\times10^{-2}$ M, TZ color = 10.0 mg $L^{-1}$, pH= 4).

*Impact of ionic power of the medium*

To check the influence of ionic power of the medium, 1 ml NaCl, KCl and $NaNO_3$ ($3.0\times10^{-3}$ mol $L^{-1}$), 1 ml TZ color (10.0 mg $L^{-1}$) solution, and Starch-capped ZnSNPs ($3.0\times10^{-2}$ mol$L^{-1}$) were blended in a volumetric flask 10 ml using distilled water. The measurement of the absorbance after (25 min) sharp in solution. The impact of ionic power based on the obtained outcomes (Figure 8) on the reaction rate was negligible [33].

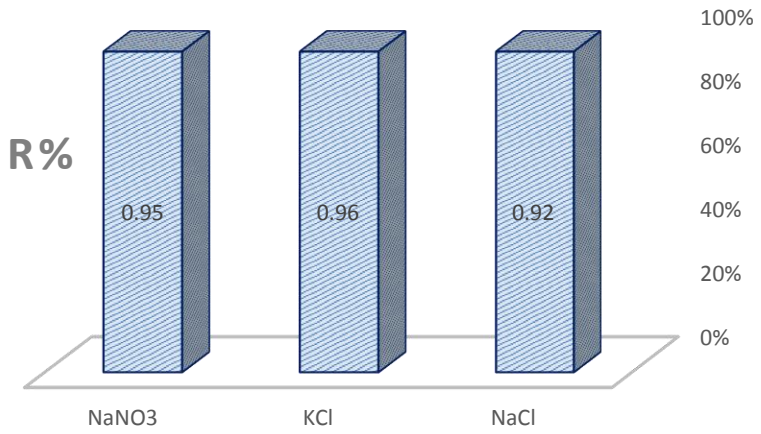


**Figure 8.** The influence of Ionic power on the reaction rate aqueous sample volume, 10 mL in 399 nm. (Starch-capped ZnSNPs, $3.0\times10^{-2}$ M, TZ color = 10.0 mg $L^{-1}$, pH= 4).

*Analytical specifications and calibration graph and reproducibility*

After optimizing the factors affecting the measurement of TZ color, the grading curve was plotted under optimized conditions (Figure 9). As shown, the absorbance intensity in the range of TZ color (0.01-20.0 mg $L^{-1}$) is linearly related to the concentration of TZ color and this error follows the equation y = 0.069x + 0.0614, where the concentration TZ color x (mg$L^{-1}$) = 0.9935 in terms of molar and correlation ($R^2$). Also, for 6 replicates, measurement of TZ color (10.0 mg $L^{-1}$), solution with optimized conditions, the relative standard deviation (R.S.D) for the response of Starch-capped ZnSNPs towards 10.0 mg $L^{-1}$ of TZ color was 1.0% and reproducibility of the response of different Starch-capped ZnSNPs was also studied. The relative standard deviation for the response of between membranes was 1.0 % (Figure 9) [34,35].

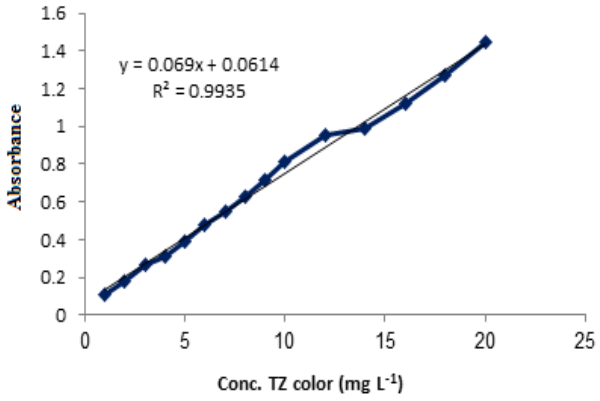


**Figure 9.** The calibration graph for TZ color, on the absorbance. (Sample volume, 10 Ml, and 399 nm).

*Optimum values of parameters*

The optimum values of the parameters are demonstrated in Table 1. The method can be used as an alternative method for TZ color measurement owing to advantages like excellent selectivity and sensitivity, low cost, simplicity, low detection limit and no need in utilizing organic harmful solvent.

**Table 1.** Investigation of method repeatability at conditions.

| Parameter | Optimum Value for TZ color |
|---|---|
| TZ color (M) | (10.0 mg $L^{-1}$) |
| Starch-capped ZnSNPs (M) | ($3.0\times10^{-2}$ M) |
| pH | 4.0 |
| Equilibration time (min) | (25 min) |
| Linear range (LDR) | (0.01-20.0 mg $L^{-1}$) |
| Detection limit (LOD) | (1.0 %) |
| Accuracy and precision | High |
| Advantages | High repeatability, sensitivity, selectivity, and wide linear range |

*Interference Studies*

After establishing the measurement method, to evaluate the selectivity of the prepared Starch-capped ZnSNPs sensor for determining the TZ color and the effect of various substances on the determination of TZ (10.0 mg $L^{-1}$), the method was tested under optimum conditions. Several representative potential interferences such as inorganic cations, anions, molecular species, and dyes were investigated individually for their effect on TZ recovery. Tolerance Limits were defined by the concentration of interface which caused <5% error in the determination of TZ [36]. The results showed that most of the other medications did not have much effect on the measurement of TZ color, and among them, compounds with a more similar structure or with

more functional groups are more disturbing. It may be related to their hydrogen interactions or the molecule of the TZ color, and thus reduce the measurement of the TZ color in the analyte sample. As exhibited in Table 2, tolerance limit was determined as the maximum concentration of the interfering substance which resulted in an error less than (±5%) for determining the TZ color. So, the selectivity of the recommended method was proven [37].

**Table. 2.** Impacts of the matrix medicaments on the retrieving of the examined TZ color (N=5).

| Foreign species | Tolerance limit (mg/L) |
|---|---|
| Sunset yellow, Glucose, Lactose, Allure red | 500 |
| Tartrate, Citrate, Oxalate, Malic acid | 300 |
| $NH_4^+$, $Mg^{2+}$, $Hg^+$, $Co^{2+}$, $Ca^{2+}$, $Cl^-$, $F^-$, $SO_4^{2-}$ | 500 |
| Vitamin B1, Vitamin $B_{12}$, Vitamin $B_6$ | 100 |

*Application of the real sample*

The complexation method was successfully applied to the determination of TZ in two different commercial food products (powered drink and powdered gelatin samples) [37]. To investigate the applicability of the proposed method, recovery experiments were performed using multiple point standard addition method. For this purpose, a known amount of TZ was spiked to the formulated preparations, and the total amount of the color was estimated (n=5). The results are summarized in Table 3 which indicate that the prepared sensor has a very good performance for determining the TZ color in food samples. Therefore, determining TZ color in samples was confirmed utilizing standard addition method. The level of the TZ color was estimated to be below the detection limit of related element. Based on the outcomes of replicating analyses for each sample, it was shown that the medication retrievals were mainly quantitative with a low RSD. The potentiality of the recommended method for the determination of trace quantities of these elements in distinct samples [38].

**Table. 3.** Retrieval of trace TZ color in Food samples after applying presented procedure (n=5).

| Samples | Added ($mg\ L^{-1}$) | Founded ($mg\ L^{-1}$) | RSD % | Recovery % |
|---|---|---|---|---|
| Smarties | 0/00 | 10/0 | 1/1 | ---- |
| | 10/00 | 20/1 | 1/3 | 101.0 |
| Noshmak | 0/00 | 2.2 | 3/7 | ---- |
| | 10/00 | 12.0 | 2/8 | 98.0 |
| Gummy candies | 0/00 | 9/9 | 3/8 | ---- |
| | 10/00 | 19/7 | 3/0 | 98.0 |
| Jelly stra wberry | 0/00 | 4/5 | 2/3 | ---- |
| | 10/00 | 14/4 | 1/8 | 99.0 |

| | | | | |
|---|---|---|---|---|
| Jell Gum | 0/00 | 6.8 | 2/5 | ---- |
| | 10/00 | 16.6 | 2/1 | 98.0 |

*Comparison of results for this work with other reported sensors*

In order to illuminate the applicability and efficiency of sensor in this work, the results are compared with those of some of the recently reported methods for the reduction of variety of materials by sensor in (Table 4). Obviously, Starch-capped ZnSNPs shows the shortest time for the reaction in comparison with literature other sensors.

**Table 4.** Comparison of results for this work with other reported sensors.

| Samples | Sensor | Time | References |
|---|---|---|---|
| Tartrazine (TZ) | XAD-16 resins | 30.0 min | [3] |
| Tartrazine (TZ) | nanosheets G-$C_3N_4$ | 2.5 min | [9] |
| Tartrazine (TZ) | Active Carbon (AC) | 15.0 min | [39] |
| Tartrazine (TZ) | $Fe3O_4$/Graphene oxide-COOH | 20.0 min | [40] |
| Tartrazine (TZ) | *Mandarin Leaves*-capped AuNPs | 7.0 min | [41] |
| Tartrazine (TZ) | Starch-capped ZnSNPs | 7.0 min | This Work |

## Conclusion

A successful analytical method for measuring TZ color was prosperously developed via utilizing a sensitized spectrophotometric method with the help of Starch-capped ZnSNPs. The method can be used as an alternative method for TZ color measurement owing to advantages like excellent selectivity, and sensitivity, low cost, simplicity, low detection limit, and no need in utilizing organic harmful solvent or extraction. The reaction was evaluated by measuring the absorption rate of TZ color, the optimum conditions. The lowest determining error TZ color could be obtained in a short time, which strongly confirms the greater contribution for the deletion of TZ color by Starch-capped ZnSNPs sensor.

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