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Design and Evaluation and Synthesis a Starch-Capped Silver NanoParticles Sensor and Determination trace Sulfacetamide Drug in the Presence Sodium borohydride in Blood and Urine Samples with Kinetic Spectrophotometric Method

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ABSTRACT

A new Kinetic Spectrophotometic method for the Determination of trace amount Sulfacetamide Drug into real samples has been described based with silver nanoparticles starch-capped sensor, by sodium borohydride. The reaction is followed spectrophotometrically by measuring the decrease in the absorbance at 347.5 nm. 1.0×10^{-2} molL^{-1} silver nanoparticles starch-capped sensor, 2.0×10^{-3} mol L^{-1} sodium borohydride at 25^{oC}, calibration graph in the range of 0.1-6.0 µg L^{-1} sulfacetamide drug. The absorbance is linear from 0.02 up to 8.0 µg L^{-1} in aqueous solution with repeatability (RSD) of 0.9% at a concentration of 6.0 µg L^{-1} and a detection limit of 2.3 µg L^{-1} by the fixed-time method of 7.0 min. The relative standard deviation for 6.0 µg L^{-1} sulfacetamide drug is %95. No serious interference was identified. The applicability of the method was demonstrated by the determination of the Sulfacetamide drug in urine and blood samples.

Keywords: Sulfacetamide Drug; Kinetic; Starch-Capped Silver NanoParticles Synthesis; Sensor; Determination; Spectrophotometic

1. INTRODUCTION

Sulfacetamide is a Sulfonamide antibiotic, that is used as a cream to treat skin infections and as eye drops to treat eye infections. On the skin it is used to treat acne and seborrheic dermatitis [1]. In cream form it is used to treat bacterial infections on the skin. It can also be used orally to treat urinary tract infections [2]. It kills the bacteria by restricting the production of folic acid, which is essential for bacterial growth [3]. It mainly inhibits the multiplication of bacteria as it acts in a competitive inhibitor. There are however also severe side effects including severe allergic reactions, like (nettle) rash, itch, tightness in chest, difficult breathing and swelling in either the face, mouth, lips or tongue. Other severe side effects include bloody or severe diarrhea, fever, joint pain, red, blistered or swollen skin and stomach pain [3,4]. In the eye, it can cause conjunctivitis. There are also life-threatening conditions which can be produced by the antibiotic, like Stevens

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Johnson syndrome and Erythema multiforme. Higher exposure can also cause unconsciousness. One case showed that sulfacetamide eyedrops can very rarely cause life-threatening skin condition [5].

Sulfacetamide has antibacterial activity and is used to control acne. Products containing sulfacetamide and sulfur (a keratolytic) are commonly promoted for the treatment of acne rosacea (rosacea with papules, pustules, or both). There are several prescription topical products containing sulfacetamide, such as foams, shampoos, cream and washes [6]. These are organic compounds containing an benzenesulfonamide moiety with an amine group attached to the benzene ring [7]. The molecular structure is $C_8H_{10}N_2O_3S$. Its scientific name is N-(4-aminophenyl) sulfonylacetamide. At room temperature, it appears as a white powder. Sulfacetamide is stable under normal temperatures and pressures. No dangerous reactions occur under known conditions of normal use [8]. It is an important bacteriostatic agent that is commonly used in human and veterinary medicine. Therefore it can accumulate in the environment (mostly surface water) [9].

In this connection, several analytical techniques such as capillary electrophoresis [10], voltammetry [10, 11], nanocomposites electrodes-based voltammetry [12, 13], high-performance liquid chromatography (HPLC) [10, 14, 15], liquid chromatography coupled with mass spectrometry (LC–MS) [15, 16] and UV–visible spectrometric methods [10, 17] have been used for the determination of one drugs in pharmaceutical and biological samples. However, these techniques are very time consuming, and required sophisticated instrumentation, and not suitable for real-time analysis. [11, 13, 14, 18]. Recently, noble metal nanoparticles-based UV–visible spectrometric methods have drawn special attention for selective and sensitive reorganization of target species (inorganic, organic and biomolecules) in various complex matrices [19, 20].

Since, Metallic NPs (Au and Ag NPs) are emerging as promising analytical colorimetric reporters for wide variety of analytes because of their intrinsically exploitable properties such as the high extinction coefficient and the distinct variation in color based on their dispersion and aggregation state [21, 22]. In recent years, metal nanoparticles (NPs) have been extensively used for detection of heavy metal ions naked-eye inspection of metal ions in environmental samples because they have size- and distance-dependent optical properties [23]. Particularly colorimetric sensors based on silver nanoparticles (AgNPs) have attracted increasing attention due to their localized surface plasmon resonance (SPR) absorption and unique optical properties [24]. The colorimetric sensing method has several advantages such as simplicity and rapidity, high sensitivity, cost-effectiveness, real-time monitoring and ease of measurement [25]. Efforts have been made to explore their attractive properties and utilize them in practical applications, such as anti-bacterial and anti-cancer therapeutics [26], diagnostics and optoelectronics [27], water disinfection [28], and other clinical/pharmaceutical applications [29]. Moreover, AgNPs are used in antimicrobial applications with proven antimicrobial characteristics of Ag^+ ions. These exceptional properties of AgNPs have enabled their use in the fields of nanomedicine, pharmacy, biosensing, and biomedical engineering [30].

In this work, Determination amount trace Sulfacetamide drug with using on Starch-Capped Silver NanoParticles Sensor, in the Presence Sodium

borohydride was investigated. A Kinetic Spectrophotometric method for the determination of sulfacetamide drug was developed. The proposed method is extremely sensitive, with higher selectivity and is a simpler procedure than the three methods mentioned above. The method was applied successfully to the determination of sulfacetamide drug in blood and urine samples.

2. EXPERIMENTAL

2.1. Instrumentation

UV–Visible Spectra were Measured by using a Maya Pro 180 Spectrophotometer (Shimadzu, Japen) at room temperature. Fourier transform infrared (FT-IR) spectra were recorded on a Perkin Elmer (FT-IR spectrum BX, Germany). A JEOL 3010 was used to take and record the transmission electron microscopy (TEM) images. For pH measurements, a Genway model 3510 pH/Ion meter with a combined glass electrode was utilized. Laboratory glassware was soaked nightlong in 10% aqua fortis solution (HNO_3). In order to control the temperature, a NBE ultrathermostat (VEB Prufgerate – WerkMedingen, Germany) was utilized.

2.2. Reagents and materials

All Chemicals including Silver nitrate ($AgNO_3$) and Sodium borohydride ($NaBH_4$) were provided from Merck Company while Sulfacetamide medication (98.0%) was purchased from India Company. Again, Hydrochloricacid and Methanol were bought from Merck Company (Merck, Darmstadt, Germany). DD H_2O (double distilled water) was used in preparation of the solutions. The ensuing shows the concentrations of the stock solutions

Ag/starch-capped, 1.0×10^{-2} mol L^{-1}

Sodium borohydride ($NaBH_4$), 2.0×10^{-3} mol L^{-1}

Sulfacetamide drug, 6.0 µg L^{-1}.

2.3. Pretreatment of Real Samples

2.3.1. Urine Sample

A 10 mL Portion of a urine sample (or a spiked urine sample) was treated with 10 mL of concentrated HNO_3 (63%) and an $HClO_4$ (70%) mixture of 2:1 in a 50 mL beaker covered with a watch glass. The content of the beaker was heated on a hot plate (100 °C 15 min, 150 °C 10 min). The watch glass was removed and the acid evaporated to dryness at 150 °C. $HClO_4$ (3 mL) was added to the resulting white residue and the mixture was heated at 160 °C to dryness. All heating was carried out under a hood while taking the necessary precautions. Five milliliters of 1 M H_2SO_4 was added, the mixture heated at 150 °C for 1 min, and the volume made up to the mark in a 50 mL volumetric flask. Aliquots (7 mL) of the resulting clear solution were analyzed according to the described procedure [31].

2.3.2. Blood Sample

Homogenized blood sample 20 mL was weighed accurately and in a 200 mL beaker was digested in the presence of an oxidizing agent with addition of 10 mL concentrated HNO_3 and 2 mL $HClO_4$ 70 % was added and heated for 1 h. The content of beaker was filtered through a Whatman No. 42 filter paper into a 250 mL calibrated flask and its pH was adjusted to desired value and diluted to mark with de-ionized water. In all of real and synthetic sample amount of Epinephrine drug was found by standard addition method [32].

2.4. Synthesis of Starch-Capped Ag NPs

Starch-Capped Ag NPs were Prepared by the Reduction of $AgNO_3$ with $NaBH_4$ as a modifier according to the method in the literature [33]. Briefly, 10.0 mL of Starch (0.1 mM) solution was added into the reaction flask that contained 90.0 mL of $AgNO_3$ (0.1 mM) solution under vigorous stirring. After 15 min, 1 mL of $NaBH_4$ (2.0

mM) was UV–visible spectrum of starch-capped Ag NPs. Inset picture show starch-capped Ag NPs. added into the above solution at room temperature and stirred for 1 h. The dark colloidal solution color was changed to bright yellow, confirming that the formation of starch-capped Ag NPs. The starch-capped Ag NPs solution was stored in the dark at 4.0 ± 2.0◦C to remain stable for several weeks.

2.5. Starch-Capped Ag NPs as Colorimetric Sensor for Detection of Sulfacetamide drug.

The Starch-Capped Ag NPs-based Colorimetric method was Evaluated for Detection of Sulfacetamide Drug 1.0 mL 6.0 µg L^{-1}) were added separately into 1.0 mL of starch-capped Ag NPs solution at pH 4.0 and the sample vials were kept for few minutes at room temperature and the color changes were recorded by using digital camera (Fig. 1). The UV–Visible absorption Spectra of the resulting solutions were recorded at different time intervals from 0 min to 7.0 min.

2.6. Procedure

A Typical Kinetic Spectrophotometric method experiment required the following steps: 1 ml of sulfacetamide drug 1.0 mL 6.0 µg L^{-1} was added to the 10 ml volumetric flask. Than 1 ml of sodium borohydride (2.0×10^{-3} mol L^{-1}) and 1 ml starch-capped AgNPs 2.5×10^{-2} mol L^{-1} was added. The spectrum of sulfacetamide drug solution (6.0 µg L^{-1}) at wavelength of 347.5 nm decreased with the addition of starch-capped AgNPs to the solution. It is exhibited in (Figure 2 and 3). As can be seen, absorbance of the acetonitrile solution of starch-capped AgNPs at wavelength of 347.5 nm decreased with the addition of sulfacetamide drug to the solution. On the other hand, the observed spectral evolution involved the formation of a well-defined isobestic point at around 347.5 nm, indicating the presence of one absorbing complex compound between starch-capped AgNPs and sulfacetamide drug. The mole ratio method was also used for estimation of the stoichiometry of the sulfacetamide drug-starch-capped AgNPs complex and a typical mole-ratio plot is represented in Fig. 4) [34]. The obtained results show that the slope of the curve changes at 1:1 [M]/ [L], that indicate the stoichiometry of the complex formed between starch-capped AgNPs and sulfacetamide drug is 1:1.

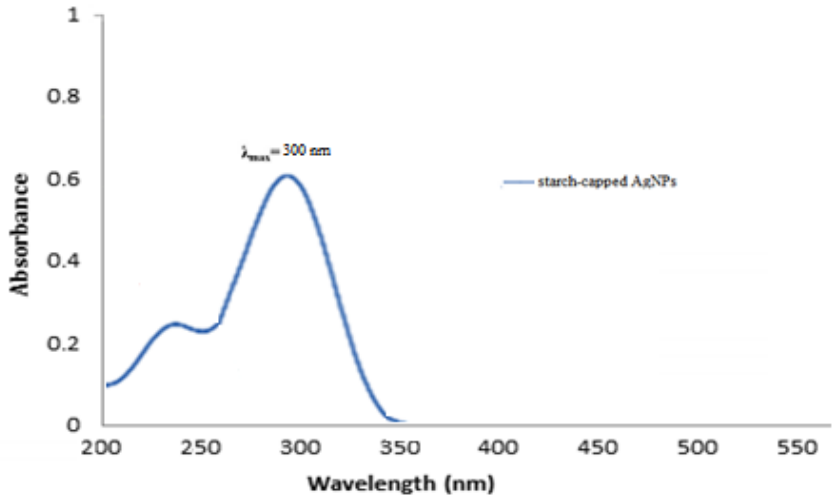

Fig. 1. The absorption spectra of product starch-capped Ag NPs.

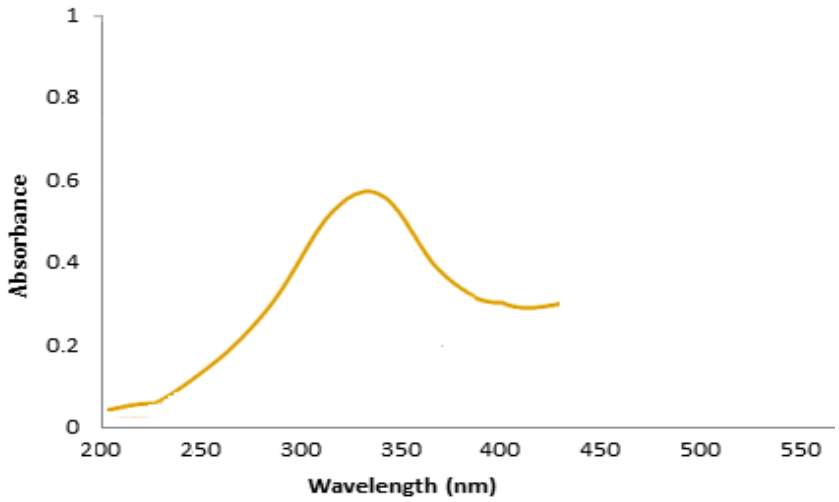

Fig. 2. The spectrum of sulfacetamide drug solution (6.0 µg L^{-1}).

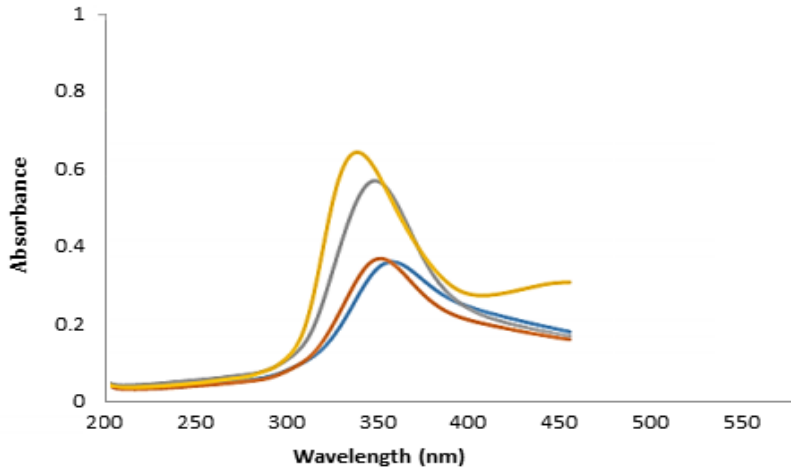

Fig. 3. The spectrum of sulfacetamide drug solution (6.0 µg L^{-1}) in acetonitrile and increasing concentration of sulfacetamide drug solution (1.0 µg L^{-1}).

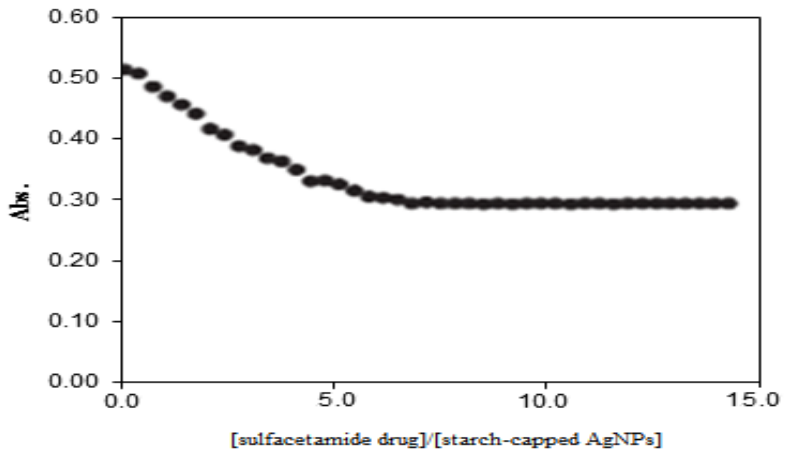

Fig. 4. Mole ratio plots of absorbance as [Sulfacetamide drug]/[starch-capped AgNPs] at 347.5 nm.

3. RESULTS AND DISCUSSION

3.1. Characterization of adsorbent

The FTIR spectrum of starch-capped AgNPs nanoparticles loaded on activated carbon (Fig. 5b.), The broad peaks at ≤900 in Ag – O, 1040-875 cm^{-1} could be assigned to C–H stretching from starch-capped AgNPs, and 1444cm^{-1} to C=O bonds. The new peak appearing at 2917 cm^{-1} corresponds to C–H stretching new peak appearing at 3422 cm^{-1} corresponds to – OH stretching [35]. The XRD pattern of the starch-capped AgNPs (Fig.5a) represents a peak at 38.07 (111), 44.26 (200), 64.43 (220) and 77.35 (311) correspond to diffractions and reflections [36, 37] As seen, the highly crystalline nature of the after functionalizing with starch-capped AgNPs is confirmed, while the high intensity of peak at 38.07 (111) shows that there has been a small amount of material in amorphous state. The observed XRD pattern indicates that the prepared starch-capped AgNPs is well-synthesized. The morphological features of the samples studied by SEM are shown in Fig.6 a,b).

TEM are shown in Fig.6 c,d) starch-capped AgNPs are observed to be smooth, homogeneous, tidy and approximately uniform in size distribution (Fig.6). Afer the surface modification with starch-capped AgNPs became rough, larger and bundled [38].

3.2. Effect of pH

In order to investigate the best pH for effective colorimetric sensing of sulfacetamide drug with starch-capped Ag NPs, we studied the UV–visible absorption spectra of starch-capped AgNPs by the addition of sulfacetamide drug at different pH ranges in 347.5 nm from 2.0 to 9.0 (Fig. 7) [39]. The absorbance measurements were expressed as absorbance difference, which was defined as the difference between the absorbance of the immobilized starch-capped AgNPs alone and the absorbance of the sulfacetamide drug- starch-capped AgNPs complex at 347.5 nm. As can be seen in Fig.7, the pH increases from 2 to 4.0 as the value of the difference in absorbance increases. At pH values more than 4, the response decreases. This phenomenon might be due to the fact that at lower pH values (pH<4), complexation is weak. At pH values higher than 3, sulfacetamide drug forms different hydroxide species which make it unable to form complex with starch-capped AgNPs [40]. Thus, pH=4 was selected for furthure studies.

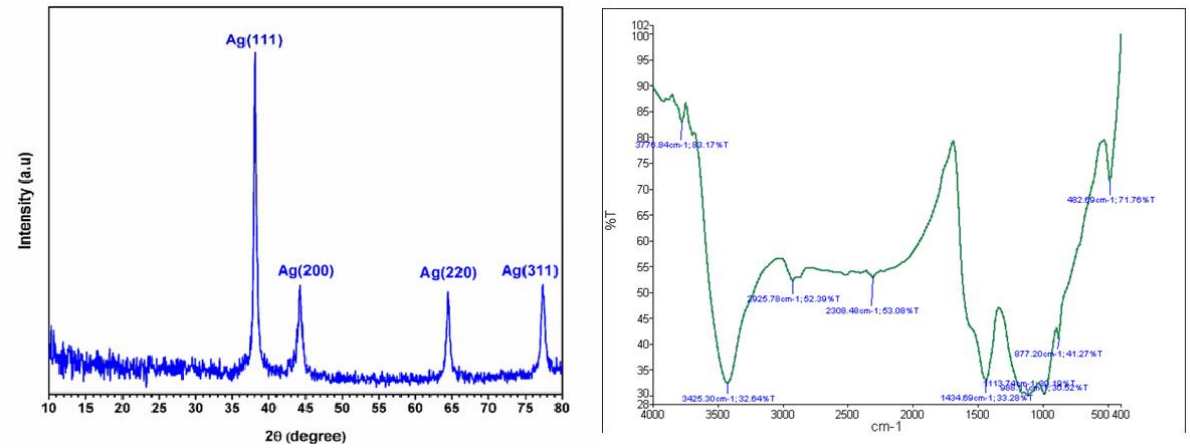

Fig. 5. (a) XRD image and (b) FT-IR transmittance spectrum of the prepared starch-capped AgNPs.

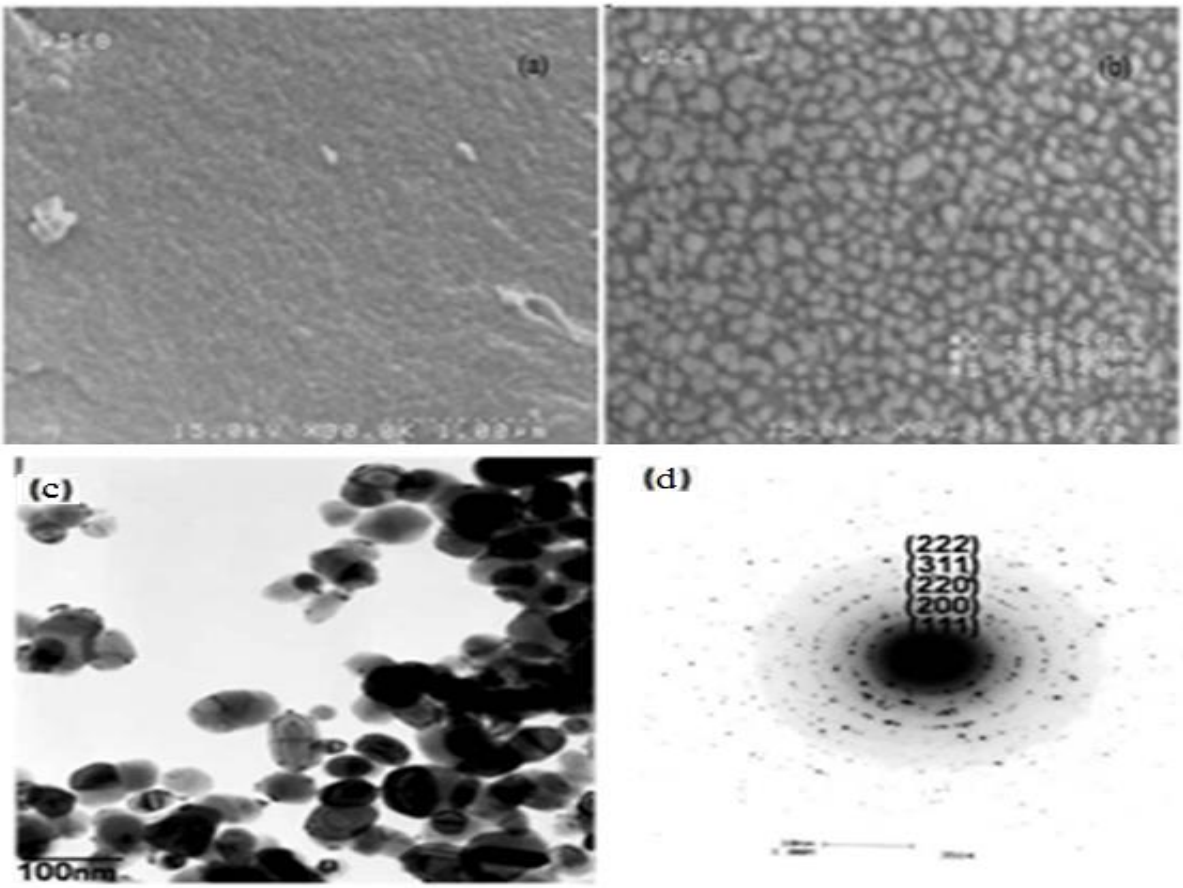

Fig. 6. The (a,b) SEM image and (c,d) TEM of the prepared starch-capped AgNPs.

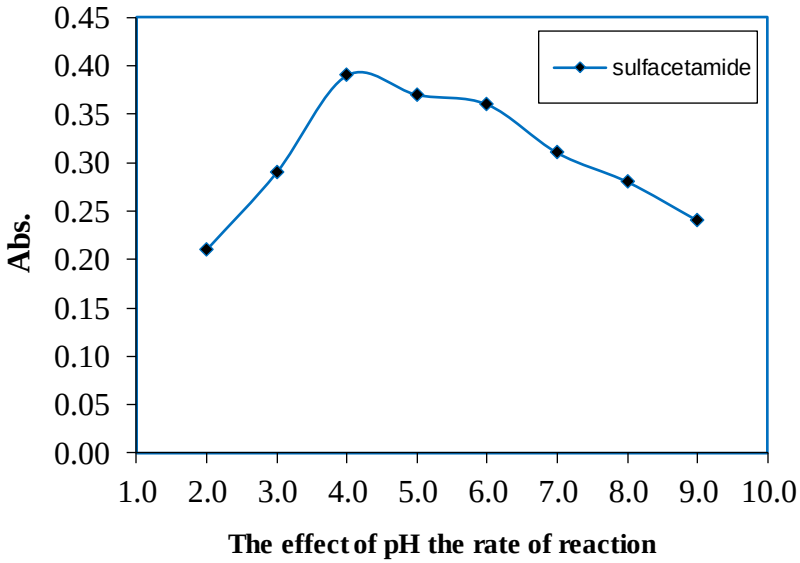

Fig. 7. The effect of sulfacetamide drug concentration on the rate of reaction

3.3. Effect of Starch-Capped AgNPs on the Reaction Rate

To inspecting the effect of starch-capped AgNPs, on the reaction rate, 1 ml sulfacetamide drug 6.0 µg L^{-1} solution, 1 ml sodium borohydride 2.0 × 10^{-3} mol L^{-1}, and 1 ml, starch-capped AgNPs, 0.5× 10^{-3} to 4.0 × 10^{-2} mol L^{-1} are added to the volumetric flask 10 ml and by adding distilled water. Absorption of solution was measured after 7.0 min. The above mentioned operation was repeated for blank solution (the solution without sulfacetamide drug). The results are shown in and (Figure 8), based on those results 1.0 × 10^{-2} mol L^{-1} was selected as the desired concentration.

3.4. Effect of Sodium borohydride Concentration on the Reaction Rate

To inspecting the effect of sodium borohydride concentration, 1 ml sulfacetamide drug 6.0 µg L^{-1} solution, 1 ml of sodium borohydride at different concentration 0.02 to 3.0×10^{-3} mol L^{-1}, and 1 ml starch-capped AgNPs, 1.0×10^{-2} mol $L^{-1}$are added to the volumetric flask 10 ml and by adding distilled water. Absorption of solution was measured after 7.0 min. The above mentioned operation was repeated for blank solution (the solution without sulfacetamide drug). Results are shown in figure 9, based on that results, sodium borohydride at a concentration of 2.0×10^{-3} mol L^{-1} was selected as the desired concentration.

3.5. Effect of Time on the Reaction Rate

As it was expressed in the method, to obtain optimum time of the reaction, 1 ml sulfacetamide drug 6.0 µg L^{-1} solution, 1 ml sodium borohydride 2.0 × 10^{-3} mol L^{-1}, and 1 ml starch-capped AgNPs, 1.0 × 10^{-2} mol L^{-1} are added to the volumetric flask 10 ml and by adding distilled water. Absorption of solution was measured in the 0-10 min interval of time. The above mentioned operation was repeated for blank solution (the solution without sulfacetamide drug). Changes in absorption based on the time at 25^{oC} centigrade degrees temperature are shown in (Figure. 10). 7.0 min was selected as the optimum time.

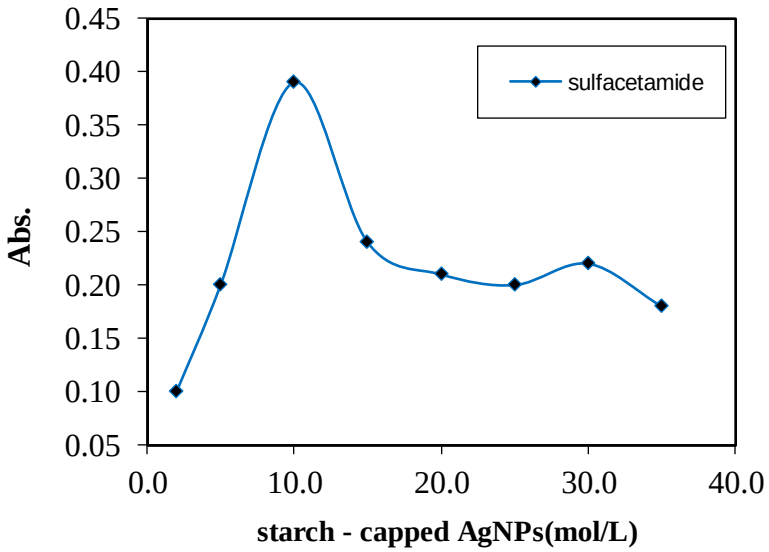

Fig. 8. The effect of starch-capped AgNPs on the reaction rate.

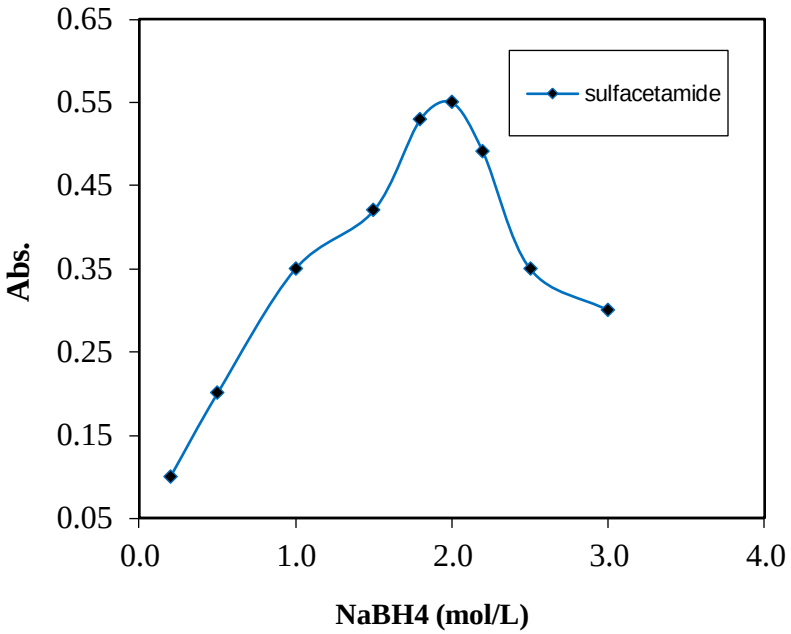

Fig. 9. The effect of sodium borohydride ($NaBH_4$) on the reaction rate.

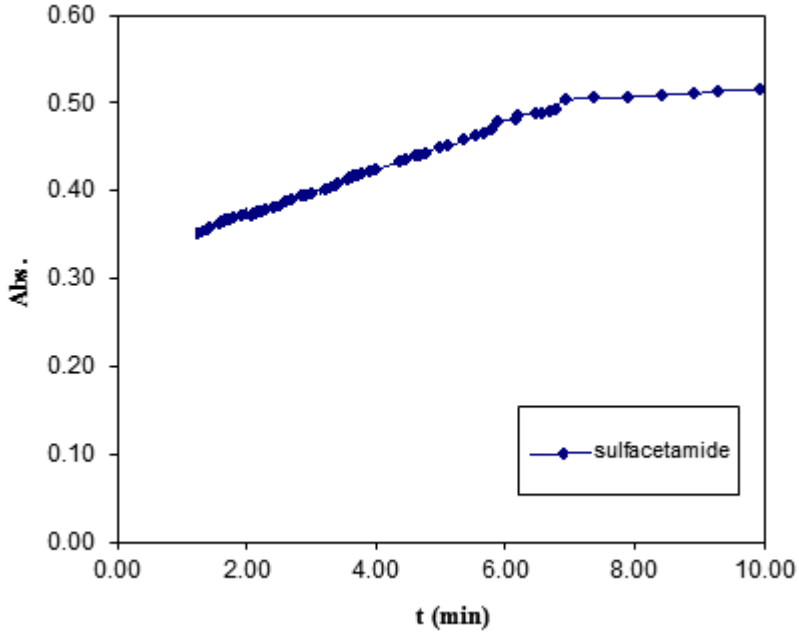

Fig. 10. The effect of time on the reaction rate.

3.6. Effect of Ionic Power of the medium was also

Inspected; Potassium bromide and Potassium nitrate were used for this purpose. 1 ml sulfacetamide drug 6.0 µg L^{-1} solution, 1 ml sodium borohydride 2.0 × 10^{-3} mol L^{-1}, and starch-capped AgNPs, 1.0×10^{-2} mol L^{-1} are added to the volumetric flask 10 ml and by adding distilled water. Absorption of solution was

measured after 7.0 min. as we can see in (figure 11), results show that the effect of ionic power on the reaction rate.

3.7. Response time

The response time of the membrane is controlled by the time required for the analyte to diffuse from the bulk of the solution to the starch-capped AgNPs interface with the indicator [41]. The response time of the present membrane was tested by recording the absorbance change at 347.5 nm from a pure buffer (pH=4) to a buffered sulfacetamide drug solution of 6.0 µg L^{-1}. The starch-capped AgNPs was found to reach 95% of the final signal at 7-10 min depending on the concentration of the sulfacetamide drug (Fig.12). In general, the response time is lower in concentrated solutions than dilute solutions.

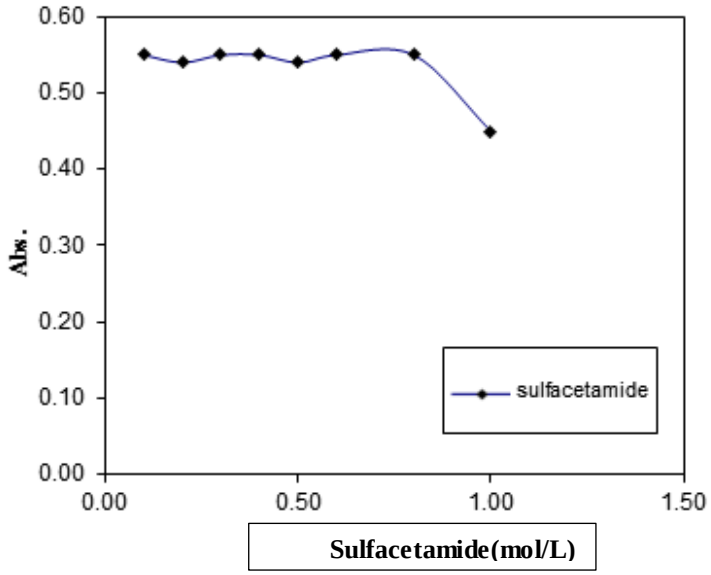

Fig. 11. The effect of ionic power on the reaction rate.

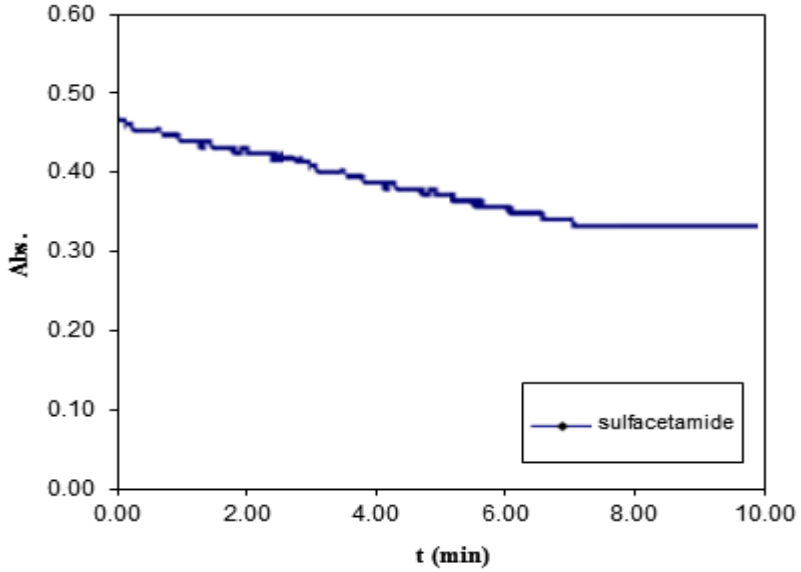

Fig.12. Typical response curve of the starch-capped AgNPs at 347.5 nm as a function of time when film was exposed to 6.0 µg L^{-1} sulfacetamide drug.

3.8.Calibration Graph and Reproducibility

These parameters of the starch-capped AgNPs in the determination of sulfacetamide drug was evaluated by repeatedly exposing the sensing phase membrane to a 0.1 µg L^{-1} sulfacetamide drug solution and a 6.0 µg L^{-1}. The repeatability was evaluated by performing seven determinations with the same standard solution of sulfacetamide drug. The relative standard deviation (R.S.D) for the response of starch-capped AgNPs towards a 6.0 µg L^{-1} of sulfacetamide drug solution was 0.9% (n=7).

The reproducibility of the response of different starch-capped AgNPs was also studied. Seven different membranes were prepared from the same batch and they were evaluated by performing the determination of 6.0 µg L^{-1} sulfacetamide drug. The relative standard deviation for the response of between membranes was a detection limit of 2.3 µg L^{-1} by the fixed-time method of 7.0 min in 347.5 nm.

3.8. Interference Studies

The accuracy and reliability of this method was evaluated by comparing the results obtained for the same samples by an method. To investigate the interference some biological coexistents frequently exist in the body fluids such as sulfacetamide drug and other drugs; were evaluated. The results estimated by spectrophotometry are much less as compare to results obtain by spectophetometric. It may be attributed to the interference of foreign drugs in the determination of sulfacetamide drug interfering drugs. These drugs may interfere with sulfacetamide drug. In order to assess the potential analytical applications of the proposed kinetic reaction, the influence of foregn drugs on the determination of sulfacetamide drug was investigated [42]. The tolerance limit was defined as the maximum concentration of the interfering substance that caused an error less than ±5% for determination of sulfacetamide drug are shown in Table1. As can be seen, the proposed method is highly selective.

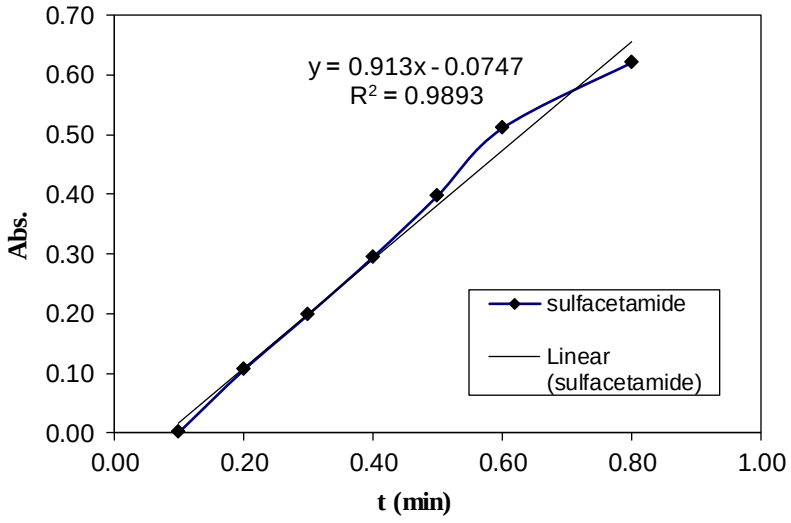

Fig. 13. Calibration graph for sulfacetamide drug.

3.9. Application

To assess the applicability of the method to real samples with different matrices, containing various amounts of drug was used. For accuracy and reliability of proposed method, spiking experiments and independent analysis were used [43]. The proposed method was applied to the determination of total sulfacetamide drug in urine and blood samples. In (Table 2) the results obtained are shown. The level of the sulfacetamide drug were found below the detection limit of related element. The results of replicate three analyses of each sample show that the drugs recoveries are almost quantitative with a low RSD [44, 45]. The recovery of spiked samples is satisfactory reasonable and was confirmed using standard addition method, which indicate the capability of the proposed method for the determination of trace amounts of these drugs in different samples.

4. CONCLUSION

The optimum value of parameters are presented in (Table 3). The proposed method offers the advantages of simplicity, selectivity and high sensitivity for the determination of sulfacetamide drug without the need for organic solvent extraction, preconcentration or pre-separation. An efficient analytical method for determining the determination sulfacetamide drug was successfully developed by using a sensitized spectrophotometric using starch-capped AgNPs. The method due advantages such as high selectivity and sensitivity, low detection limit, simplicity, low cost and no need to extraction and using organic harmful solvent with respect to previously reported methods is an alternative method for sulfacetamide drug determination.

Table 1. Effects of the matrix drugs on the recoveries of the examined sulfacetamide drug (N=3)

drugs	Effects of the matrix drugs (mg L^{-1})
Amoxicillin, Ampicilline, Acetominophene, Cortisone, Cyclosporine	1000
Tramadol, Metadone	750
Naratriptan, Rizatriptan, Sumatriptan, and Zolmitriptan	500
Epynephrine	100

Table 2. Recovery of trace sulfacetamide drug from urine and blood sample after application of presented procedure (N=3)

sample	Added (µg mL^{-1})	Founded (µg mL^{-1})	RSD %	Recovery %
urine	0/00	0/41	1/1	---
	0/15	0/42	2/0	99
blood	0/00	0/35	2/8	
	0/15	0/36		103

Table 3. Investigation of method repeatability at conditions according to Table 3

Parameter	Optimum Value for sulfacetamide drug
Sulfacetamide drug (µg L^{-1})	6.0 µg L^{-1}
starch-capped AgNPs (mol L^{-1})	1.0×10^{-2} M
concentration $NaBH_4$(mol L^{-1})	2.0×10^{-3} M
pH	4.0
Equilibration time (min)	7.0

Parameter	Optimum Value for sulfacetamide drug
(R.S.D)	% 0.9
Detection limit ($\mu g\ L^{-1}$)	2.3
Accuracy and precision	High
Advantages	High repeatability, sensitivity, selectivity, wide linear range and no need to organic solvent
Disadvantages	Do not preconcentrate

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