

Effect of pH in Metal Salts Ability Study Towards Anionic Surfactant Detection

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Abstract: Extensive used of surfactants mainly anionic surfactant (AS) in cleaning products such as detergent, soap and shampoo greatly affect the water quality when released in the environment. A simple and rapid spectrophotometric method in detecting AS in water is aimed to replace the current conventional methylene blue active substance (MBAS) method that is tedious, time-consuming and requires high amount of toxic solvent. In this study, Copper (II) Bromide metal salts was chosen because of its colour, solubility in water, stability and good performance compared to other metal salts. The interaction between selected Copper (II) Bromide metal salts, Sodium Dodecyl Sulfate (SDS) and Triethylamine (TEA) with the influence of pH is used as the method to obtain absorbance value at 650 nm wavelength and to construct the calibration curve. The R^2 of 0.9921 was obtained from linearity graph of absorbance against concentration together with the linear equation, $y = 0.0812x + 0.05$ that was used to find the final concentration for adjusted pH 3.3 to pH 8 in different concentration of SDS by substituting absorbance value from calibration curve. This pH experiment's selectivity suggests that Copper (II) bromide might be the only metal ions that can interact with AS in the presence of TEA for the pH condition. The positive values of UV adsorption implied the suitability of working pH at 5.6 to 6.8 for Copper (II) Bromide and surfactant response.

Keywords: Anionic Surfactants, Metal Salts, Sodium Dodecyl Sulphate, Spectrophotometry, Absorbance

1. Introduction

Anionic surfactant (AS) account for roughly half of the global production mainly in cleaning products such as shampoos, dishwashing liquids and detergent. Every day, massive amounts of surfactants, particularly AS, end up dispersed in various environmental compartments including soil, water, and sediment. AS is mostly degraded in wastewater treatment plants, but some end up in surface waters, soil, or sediment. AS accumulation in sewage sludge treatment is also a source of concern as the accumulated surfactants with high concentration can inhibit sewage sludge microorganisms and hinder the operation to removes pollutants and breaks down sewage in wastewater treatment plant

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(WWTP). Ecosystem health and environmental sustainability are greatly impacted by the perseverance of the transformed products in the environment [1]. AS may enter drinking water through MWTPs, posing a risk to the health of people, animals, and aquatic life [2]. Plus, AS also can cause serious pollution on environment and plant.

Copper (II) Bromide metal salt has the capability to be used as an agent for ion-pairing with AS such as Sodium Dodecyl Sulfate (SDS) as they pass the criteria for cationic dyes. The main criterion for this method is that the adsorbent must be sufficiently soluble in water to allow for easy disposal of excess adsorbent, and that the ion-association complex must be highly extractable in a suitable organic solvent [3]. Besides that, the dye should have a cationic ionic nature and display an infinite shade range, very bright, have strong tinctorial properties, and many of the colors have fluorescent properties. When glacial acetic acid is present, the dyes should dissolve very well in water [4]. The use of various cationic dyes as counter ions other than methylene blue [5], [6] includes acridine orange (ACO) [3], Rhodamine 6G [7], n-hexane [8] and crystal violet (CV) [9], are referred in this study.

Spectrophotometry method is a common and low-cost method for measuring light absorption or the concentration of chemicals in a solution and can also be utilized to estimate the amount of a known chemical substance. The essence of this interaction is determined by the material's physical properties, such as whether it is clean or tainted, transparent or opaque, smooth, or rough, thin, or thick. The material's significant physical properties can therefore be quantified using spectrophotometric measurements. Spectrophotometric measurements of the material's phenomenological optical properties include spectral reflectance, transmittance, absorptance, emittance, scattering, and fluorescence [10]. Spectrophotometry method employs a light beam that passes through the sample, with each compound in the solution absorbing or transmitting light of a specific wavelength. Light absorption will increase, and light transmission will decrease if the concentration of a substance increases [11].

Based [5] in their research proposed a simplify spectrophotometric method using methylene blue (MB) for determining AS which makes it possible to use a smaller volume of chloroform and to exclude the stages of filtration. A very simple, rapid, reliable spectrophotometric method was developed by [3] where anionic surfactant SDS forms a yellow complex with the cationic dye acridine orange (ACO), and toluene is used to extract the complex. Although, all of these methods still used the toxic solvents such as toluene and chloroform for the extraction of ion associates but each methods introduces modify method that can decrease the disadvantages of official method. There is also other spectrophotometric determination method of AS that are successfully produced without solvent extraction with the use of cyanine [12] and brilliant green (BG) [13] as cationic dyes. In this study, the ability of Copper (II) Bromide (CuBr_2) metal salt with the addition of Triethylamine (TEA) to detect the presence of SDS through the absorbance value using the spectrophotometry method is expected to give different results compared to past research. This is because, the spectrophotometry method does not require to use toxic solvents such as toluene and chloroform that is used in standard method by [7].

The effect of pH in different SDS concentration interaction with Copper (II) Bromide metal salts and TEA are studied. Concentration and pH are among the factors that influence surfactant detection in spectrophotometry. The absorbance value is influenced by pH as study by [12] which show the comparison between pH against absorbance in both absence and presence of SDBS. The results indicate that the absorbance with overall range of pH in the absence of SDBS is higher than with presence of SDBS. This is because, the maximum absorbance of a solution increases as the pH of the solution rises because more protonated ions are present in the solution. Apart from that, [9] examined the impact of the buffer solution on the extraction of ion pairs by adjusting pH from 4 to 9 where pH 7.9 to 8.1 being the highest absorbance. Below pH 7.9, the absorbance drops, and this suggests that it might be due to the AS activity, which is ideal in alkaline medium.

Besides that, a high concentration of AS in a sample can also result in high absorbance value as the absorbance of a substance is proportional to its concentration. This is because the quantity of molecules that light interacts with has an impact on the amount of light that is absorbed. Concentrated solutions absorb more light because they have more molecules interacting with the light entering them. Diluted solutions have lower absorbance because there are fewer molecules available to interact with light.

Study on the impact of the concentration of Rhodamine 6G [7] and Brilliant Green (BG) [13] on the absorbance demonstrate how the absorbance rises as the concentration does.

Therefore, the interaction of Copper (II) Bromide, SDS and TEA in different range of pH using a simple and rapid spectrophotometric method in detecting AS in water which is aimed to replace the current conventional methylene blue active substance (MBAS) method that is tedious, time-consuming and requires high amount of toxic solvent is further study.

2. Methodology

The flowchart of study shown in the Figure 1 is the summary of methodology that was carried out in this study.

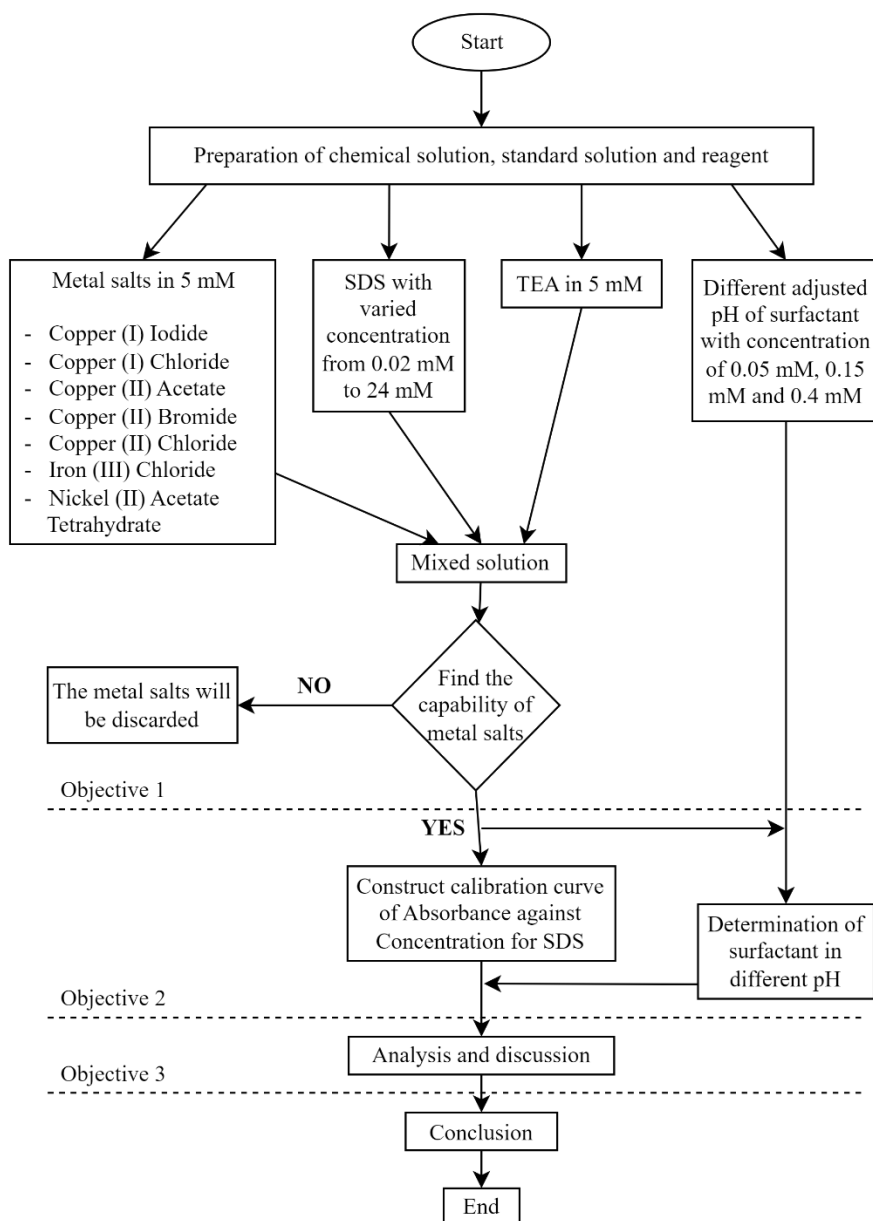


Figure 1: Flowchart of study

2.2 Preparation of SDS in different concentration

The concentration of 1 mM SDS stock solution was prepared in a 1000 mL volumetric flask by weighing 0.28838 g SDS powder as calculated using the Equation 1 and Equation 2. Next, the weighed

SDS powder was added into the volumetric flask with deionized water (DI) until it reached the calibration mark. The volumetric flask was carefully shaken to make sure the solution was mix evenly. The stock solution of 1 mM concentration of SDS were then diluted into 100 ml volumetric flasks to prepare 0.04 mM, 0.05 mM, 0.06 mM, 0.1 mM, 0.15 mM, 0.2 mM, and 0.5 mM concentration of SDS using Equation 3.

Equation to calculate the number of moles,

$$n = CV \quad \text{Eq. 1}$$

Where C is the concentration (M) and V is the volume (L)

Equation to calculate the mass needed,

$$\text{Mass (g)} = \text{Molar Mass} \left(\frac{\text{g}}{\text{mol}} \right) \times \text{Number of Moles (mol)} \quad \text{Eq. 2}$$

Equation to calculate volume for dilution,

$$M_1V_1 = M_2V_2 \quad \text{Eq. 3}$$

Where M_1 is the initial concentration (mM), V_1 is the initial volume (mL), M_2 is the final concentration (mM) and V_2 is the final volume (mL).

2.3 Preparation of Copper (II) Bromide (CuBr_2) metal salt stock solution

The 5 mM concentration of CuBr_2 stock solution was prepared by weighing 0.1137 g CuBr_2 black metal salt. The weighed CuBr_2 metal was then added into 100 ml volumetric flask with DI until it reached the calibration mark. Light blue coloured solution was observed after the volumetric flask containing the mix of CuBr_2 metal and DI was shaken to make sure the solution was mix evenly.

2.4 Preparation of Triethylamine (TEA) stock solution

The stock solution of 5mM Triethylamine (TEA) concentration was prepared by pipetting 0.173 mL of TEA liquid into 250 ml volumetric flask and DI was added until it reached the calibrated mark. The solution containing TEA liquid and DI were shaken thoroughly.

2.5 Determination of absorbance values for reaction between CuBr_2 , SDS and TEA

The volume of 1 mL of 5 mM CuBr_2 and 1 mL of 0.02 mM SDS were pipetted into a cuvette and mixed using dropper. Next, 1 mL of 5mM TEA was added into the solution in the cuvette and mixed with dropper. The cuvette was wiped with soft tissue before it was put inside the UV-Vis Spectrophotometer to determine the absorbance values for wavelength range from 400 nm to 800 nm. The graph of absorbance against wavelength were obtained and the results were recorded. The tests were repeated with 0.04 mM, 0.05 mM, 0.06 mM, 0.08 mM, 0.1 mM, 0.13 mM, 0.15 mM, 0.16 mM, 0.2 mM, 0.25 mM, 0.3 mM, 0.4 mM, and 0.5 mM concentration of SDS.

2.6 Preparation of hydrochloric acid (HCl) and sodium hydroxide (NaOH)

The concentration of 0.5 M NaOH was prepared. 2 g of NaOH pellets were weighed on analytical balance and were dissolved in 80 mL of deionised water in a 100 mL volumetric flask and shaken. When NaOH solution cooled down, DI was added again until it reached the calibration mark and shaken. The solution was transferred to a glass bottle, sealed with parafilm, and labelled. The dissolution of sodium hydroxide in water is a highly exothermic reaction, stirring should commence immediately upon addition of the water to avoid localised hot spots in the container. Diluted concentration of 0.5 M HCl 33% was prepared. 10.17 M of HCl was diluted into 0.5 M concentration by adding 20 mL DI

before 4.92 mL HCl was pipetted little by little into 100 mL volumetric flask and shaken. Next, the DI was added again until it reached the calibration mark and shaken. The solution was transferred to a glass bottle, sealed with parafilm, and labelled.

2.7 Determination of absorbance value for reaction between CuBr_2 , SDS and TEA in different pH

Five set of each 0.05 mM, 0.15 mM, and 0.4 mM SDS concentration were diluted from 1 mM stock solution and adjusted the pH by pipetting certain amount of 0.5 M HCl and 0.5 M NaOH into the 100 mL volumetric flask. The five set should consist of adjusted pH range 4 to 9 where 2 sets need to be in acidic pH, another 2 sets in alkaline pH and remaining one set was not adjusted the pH. This to construct smooth graph for the results obtained. The volumetric flasks were first filled with DI not until the calibrated line after 1 mM SDS stock solution were pipetted into them. Next, all the set were pipetted with 0.5 M HCl and 0.5 M NaOH with the first set was added with 50 μL HCl and 50 μL NaOH, the second set was added with 30 μL HCl and 30 μL NaOH, the third set was added with 61 μL HCl and 68 μL NaOH, the fourth set was added with 40 μL HCl and 50 μL NaOH and the last set were not adjusted the pH and only contained initial concentration. All the volumetric flasks were shaken carefully before the DI were added again until it reached the calibration line. The pH adjusted solution for all set were shaken carefully again and transferred to 50 mL beakers before they were pipetted for 1 mL and mixed with 1 mL CuBr_2 and 1 mL TEA in the cuvette and run with UV-Vis Spectrophotometer to measure the absorbance value from 400 nm to 800 nm wavelength. The remaining pH adjusted SDS solution in the beakers were measured the final pH using Hanna portable pH meter and recorded the value.

3. Results and Discussion

3.1 Selection and determination of metal salts capability for this study

Among the seven shortlisted metal salts which are Copper (I) Chloride, Copper (II) Chloride, Copper (I) Iodide, Copper (II) Bromide, Copper (II) Acetate, Nickel (II) Acetate Tetrahydrate and Iron (III) Chloride only Nickel (II) Acetate, Copper (II) Acetate, Copper (II) Chloride, and Copper (II) Bromide were further used in this study because of their colour and solubility in water. The four metal salts were undergone chemical mixing with different concentration of SDS and TEA before they were measured with UV-Vis Spectrophotometer to measure the absorbance values at wavelength 400 nm to 800 nm.

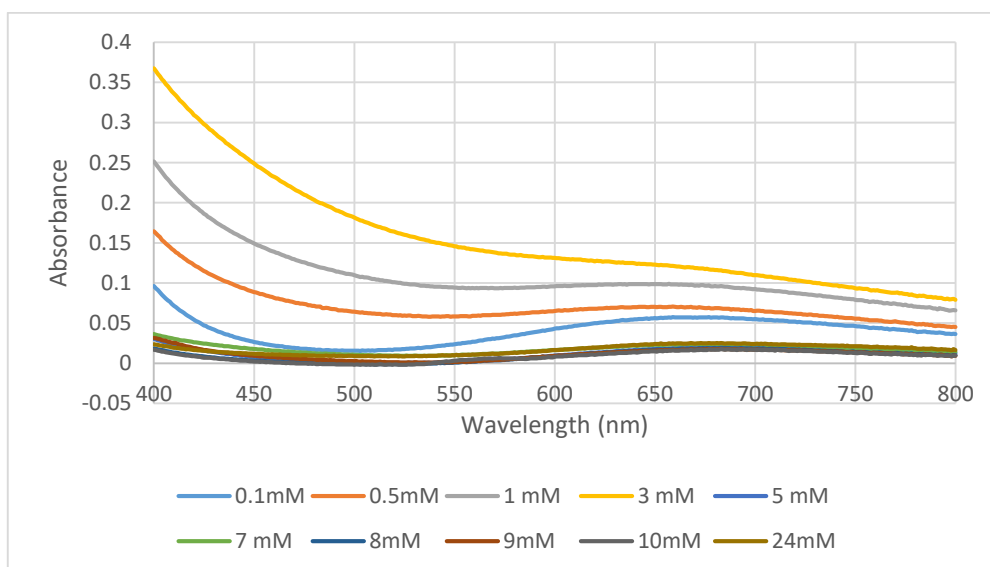


Figure 2: Graph of Absorbance against Wavelength for Copper (II) Bromide

Based on the graphs plotted in Figure 2, the curve shape of graph 0.1 mM, 0.5 mM, 1 mM, and 3 mM SDS concentration have the same curve shape and arranged in increasing manner while for other SDS concentrations such as 7 mM, 8 mM, 9 mM, 10 mM and 24 mM graphs are overlapping with each other and have slightly different curve shape. 3 mM SDS concentration have the highest absorbance

among other concentration and are positioned above others. All the graphs show decrease-increase-decrease pattern with the increasing wavelength.

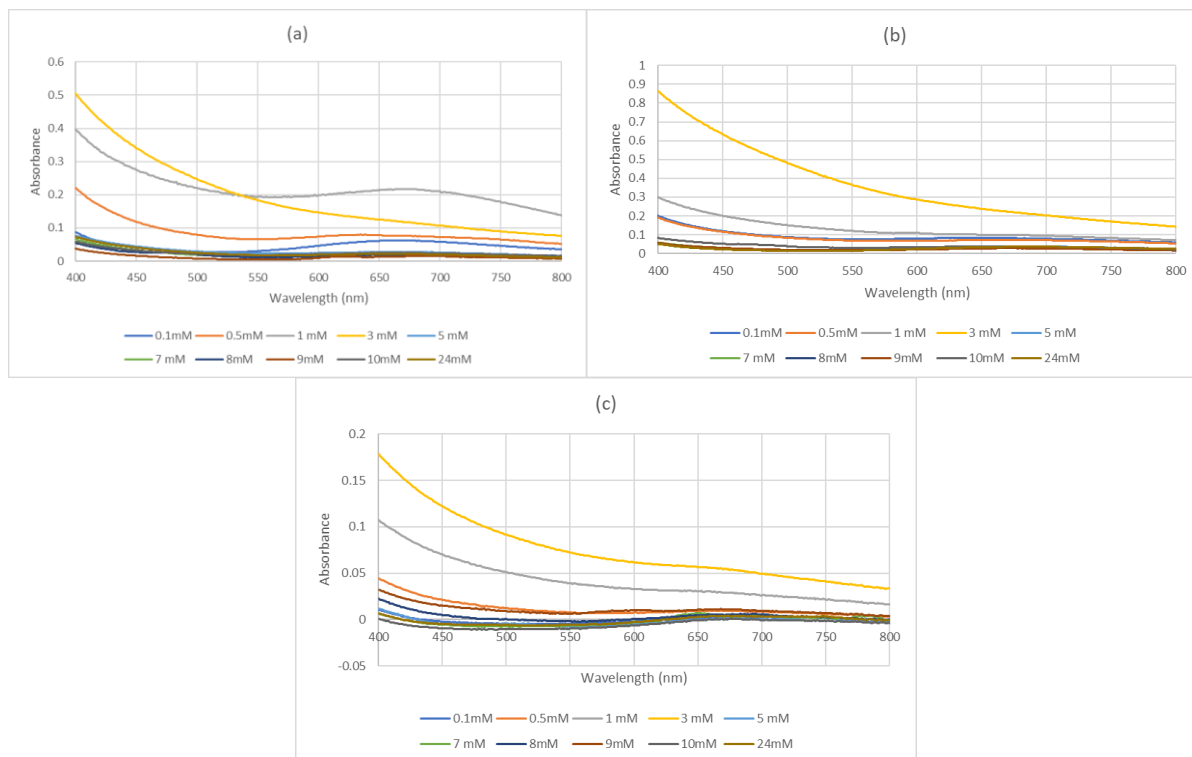


Figure 3: Graph of Absorbance against Wavelength for (a) Copper (II) Chloride, (b) Copper (II) Acetate, and (c) Nickel (II) Acetate Tetrahydrate

The graph of absorbance against wavelength range from 400 nm to 800 nm were plotted for all metal salts including Copper (II) Bromide, Copper (II) Chloride, Copper (II) Acetate and Nickel (II) Acetate Tetrahydrate. The results from graph plotted for each metal salts were compared, identify and selection of the most stable metal salts are done. When compared to other metal salts graphs in Figure 3, it can be concluded that Copper (II) Bromide metal salts graph is in Figure 2 is the most stable, smooth, and consistent graph. Therefore, Copper (II) Bromide was selected as the representative metal salt used to detect the presence of anionic surfactant in this study.

3.2 Standard calibration curve of SDS reaction with Copper (II) Bromide and TEA

After the samples were ran with UV-Vis Spectrophotometer, the calibration graph as shown in Figure 4 were plotted for absorbance against 400 nm-800 nm wavelength for SDS concentration range from 0.02 mM to 0.5 mM.

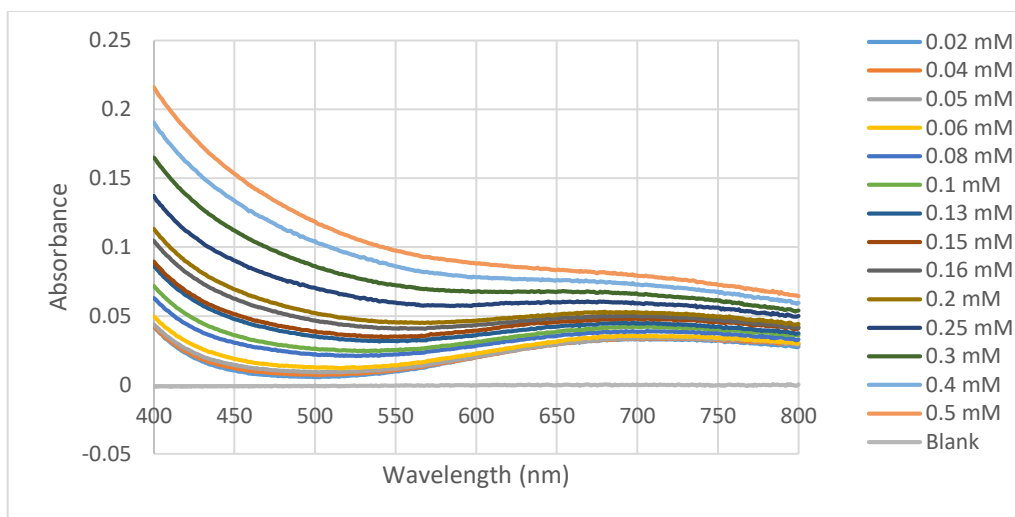


Figure 4: Graph of Absorbance vs Wavelength for Copper (II) Bromide

The smooth and stable calibration graph where all SDS concentration were systematically arranged and increasing from low to high concentration for the absorbance vs wavelength graph of Copper (II) Bromide response towards SDS and TEA. The graphs for all concentration involved start with the pattern of decreasing trend and creating the curve after increasing back at wavelength range approximately 600 nm to 700 nm and decreasing back at the end of the graph. In this study, only seven SDS concentrations as shown in Table 1 were selected from the calibration curve range in Figure 4 to be used for the study of Copper (II) Bromide response towards SDS and TEA.

As far as knowledge is concerned, none of the wavelength of Copper (II) Bromide and SDS in the presence of TEA was reported in the literature. Therefore, the chosen wavelength in this study was based on the point that varied significantly with the concentration change. In this case, 650 nm was selected as the wavelength in this study because there are slightly increased in peak around 650 nm to 690 nm wavelength of the graph shown in Figure 4.

Table 1: SDS concentration and absorbance value at 650 nm wavelength

Concentration (mM)	Absorbance
0.04	0.029298
0.05	0.029582
0.06	0.031203
0.1	0.038751
0.15	0.045494
0.2	0.050982
0.5	0.083588

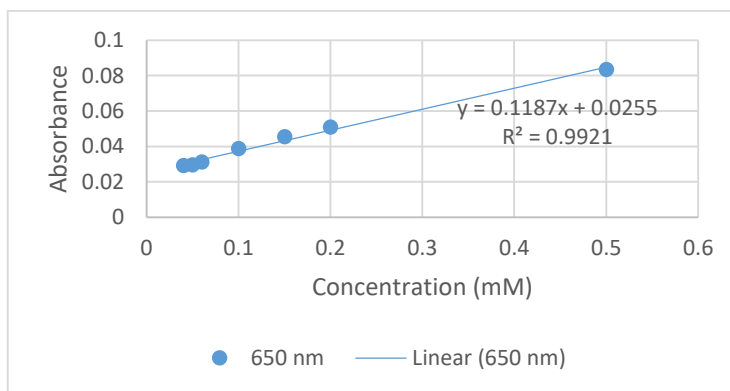


Figure 5: Absorbance vs Concentration of SDS for Copper (II) Bromide

The linearity graph for absorbance against concentration at 650 nm wavelength in Figure 5 shows a good linearity range of 0.04 mM–0.5 mM SDS concentration in an aqueous solution mixed with Copper (II) Bromide and TEA where the linear equation obtained from the base fit line produced is $y = 0.1187x + 0.0255$ and the R^2 value obtained from graph is 0.9921 which was close enough to obtain 1.

3.3 Copper (II) bromide response towards different pH of SDS concentration

To investigate the interaction and influence of pH parameter with SDS concentration, three different SDS concentration of 0.05 mM, 0.15 mM and 0.4 mM were chosen based on the calibration graph where each concentration was adjusted to five different pH by adding HCl and NaOH. Because of the difficulty to adjust and set the number of derived pH due to limited sample and sources as well as pH instability of instrumentalist, therefore, pH was only determined by volume of HCl and NaOH.

However, although the volume of NaOH and HCl used in this adjusted method were constantly mixed in the sample, the pH was observed to changed quite significant among samples. This is due to the low-quality DI which have inconsistent pH that was used in this experiment to dilute the SDS. DI can quickly adsorb carbon dioxide (CO_2) and produce carbonic acid (H_2CO_3) when exposed to air, which can cause neutral water with a pH of 7.0 to become as acidic as pH 5.6 [14]. This theory might be proven as the cause of inconsistent pH of SDS after adjusted with HCl and NaOH because of the pH recorded for initial concentration without adjusting pH of 0.05 mM, 0.15 mM and 0.4 mM were in acidic condition which were pH 5.4, pH 5.1 and pH 6.1 respectively. The pH scale is logarithmic, so even slight change in pH can cause very significant change in the chemistry.

The final concentrations were obtained in order to get the optimum pH range needed for pre-preparation of SDS for future research. This range of pH value will aid future researcher in their study. From the linear equation in Figure 4 graph, x value which is the actual concentration (mM) were calculated for each pH by substituting y with the absorbance value obtained from all pH graphs into the linear Equation 4. All data obtained were tabulated and arranged accordingly in Table 2.

$$y = 0.1187x + 0.0255 \quad \text{Eq. 4}$$

Table 2: Final pH for each SDS concentration after added with HCl and NaOH

Prepared Concentration (mM)	pH	Absorbance, y (At 650 nm)	Final concentration, x (mM)
0.05	pH 5.4	-0.025	-0.43
	pH 5.6	0.029	0.03
	pH 6.3	0.031	0.05
	pH 6.4	0.030	0.04
	pH 8.0	-0.025	-0.43
0.15	pH 5.1	-0.015	-0.34
	pH 5.8	0.046	0.17
	pH 6.1	0.047	0.18
	pH 7.8	-0.0175	-0.36
	pH 8.6	-0.020	-0.38
0.4	pH 3.3	-0.023	-0.41
	pH 5.5	-0.016	-0.35
	pH 5.9	0.075	0.42
	pH 6.1	0.061	0.30
	pH 6.8	0.065	0.33

The data tabulated in Table 2 indicates that all positive values obtained for final concentration are the indicator for optimum pH needs in the pre-preparation of SDS sample before conducting any

experiment in the future to determine the presence of SDS in synthetic water. The optimum pH range for 0.05 mM, 0.015 mM, and 0.4 mM SDS concentration is between pH 5.6 to pH 6.8. Therefore, the SDS sample prepared must be within these range because when the UV-Vis Spectrophotometer is run with SDS sample that is not in the range of optimum pH obtained from this study, the reading will give negative value. However, the validity of this method cannot be proven as the triplicate testing of samples cannot be done in this study because of factors such as constrain of chemical and time and this might affect the result obtained for this study.

3.4 Formation of ion pairing

The formation of ion pairing between Copper (II) Bromide metal salt ion and SDS ion might influence high absorbance value for optimum pH 5.6 to pH 6.8 obtained from this study. Theoretically there are three possible ion formations results based on the mechanism and binding of interaction that happened between Copper (II) Bromide metal salt, TEA and SDS.

- A. Surfactant–metal complex in Figure 6 where a coordination complex (containing a central metal ion surrounded by ligands coordinated to the metal). In these surfactants, the entity containing the central metal ion affinity toward metal ion.

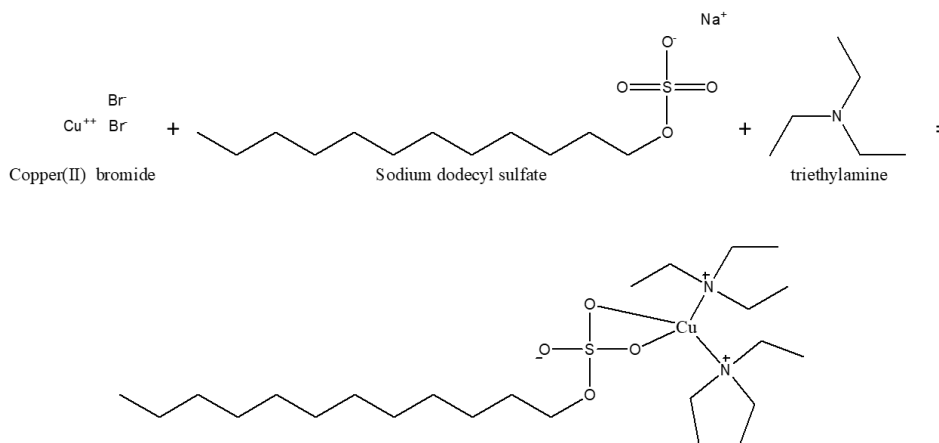


Figure 6: Formation A for pairing of ions between Copper (II) Bromide, SDS and TEA

- B. Interaction can be tail hydrophobic-hydrophobic interaction between triethyl and hydrophobic tail of SDS in Figure 7. A coordination complex with a central metal ion and ligands that are coordinated to the metal is present in the surfactant-metal complex. In these surfactants, the hydrophobic component of one or more of the ligands serves as the tail part, and the entity containing the central metal ion and its primary coordination sphere serves as the head group.

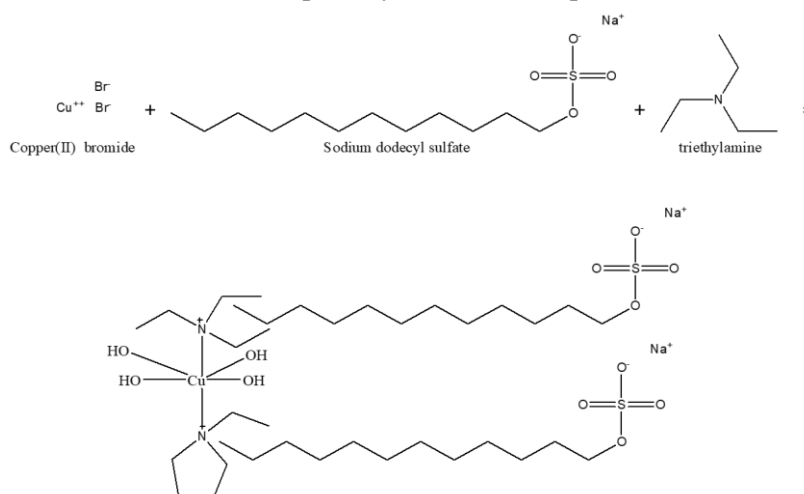


Figure 7: Formation B for pairing of ions between Copper (II) Bromide, SDS and TEA

- C. Interaction can be head electrostatic interaction between positive charge of triethylamine and negative SDS.

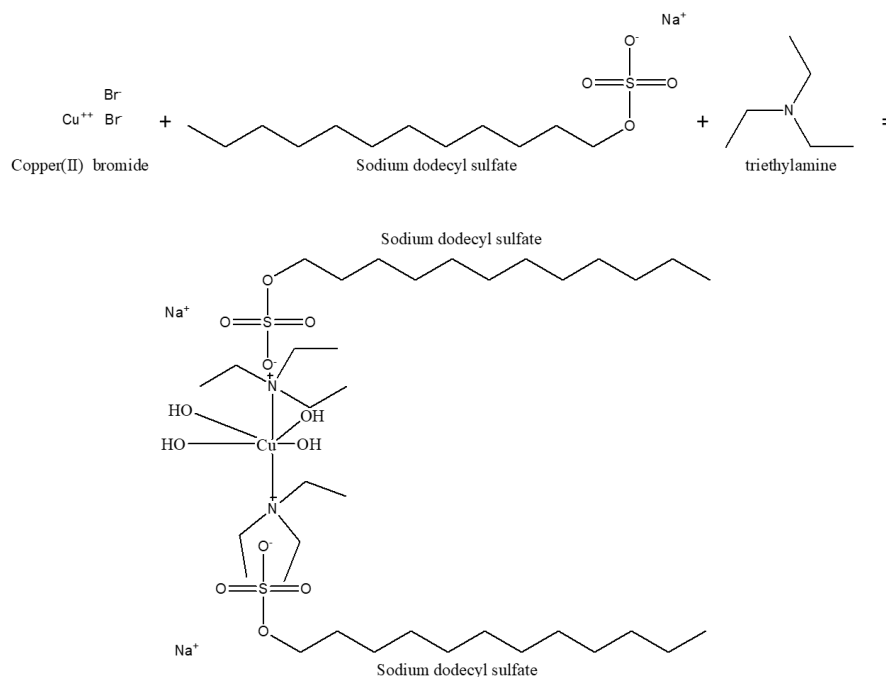


Figure 8: Formation C for pairing of ions between Copper (II) Bromide, SDS and TEA

Based on the ion's formation, the binding of protons to SDS might theoretically, inhibits the binding of metal complex to SDS when the pH is in the range of 2 to 4. When the pH ranged from 10 to 12, the interaction of hydroxyl groups with metal complex might suppressed the binding of metal complex to SDS. Study by [13] indicates that the formation of ion pair's absorption is highest at pH 6 based on the graph of absorbance against pH for ion pairing in their study. The theory might correlate for the results obtained in this study as optimum pH 5.6 to pH 6.8 are approximately near to pH 6 which is the highest pH among the pH range of 4 until 9 in their study for the formation of ion pairing. Therefore, high absorbance value might be influence by pH as the formation of ion pairing is the highest within optimum pH 5.6 to pH 6.8.

4. Conclusion

In a conclusion, the ability of metal salts including Copper (II) Bromide which was selected as the metal salt for this study because of its stability, good interaction, and response with SDS from different concentration was determined. The smooth calibration curve was constructed for Copper (II) Bromide in response with SDS and TEA. The R^2 of 0.9921 obtained was near to 1 and this indicate that good linearity graph was plotted for absorbance against concentration graph at wavelength of 650 nm which was determined from the peak absorbance of calibration curve. Additionally, the effect of pH on the Copper (II) Bromide response towards anionic surfactant in synthetic water sample were determine and the results of optimum pH for pre preparation of SDS sample obtained from final concentration results of calculation from linear graph at 650 nm absorbance wavelength in this study which is in the range of pH 5.6 to pH 6.8. It is found that there is correlation on the effect of pH on the formation of ion pairing between Copper (II) Bromide ions and SDS ions which is investigated to influence the absorbance value. Further investigation is suggested to be conducted to confirm the validity of the method conducted and findings obtained in this study. There are some improvement and recommendation for this study to produce the best result and high-quality research. Firstly, because of the pH parameter conducted in this study tends to be sensitive in pH, any chemical in the form of liquid such as DI need to be checked properly the pH before used as small changes in pH might influence and disrupt the whole results obtained. Apart from that, it is suggested for pH to be in discrete reading and uniform to all sample. This is to conclude the accurate pH effect to the solution as all changes due to metal ion,

surfactant and OH^- or H^+ will be considered. Lastly, it is suggested that the pH testing to be done in triplicate to obtain accurate results for this study.

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