HYDROGEN
JURNAL KEPENDIDIKAN KIMIA
https://e-journal.undikma.ac.id/index.php/hydrogen/index

June 2024. Vol 12 (No) 3
e-ISSN: 2656-3061
p-ISSN: 2338-6487
pp 474-486

Synthesis of Colorimetric Sensor for Cyanide Detection with Iron(III) Chloride reagent Using Sol Gel Method with Smarthphone Combination

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Article History
Received : 12-06-2024
Revised : 24-06-2024
Published: 29-06-2024

Keywords: *Colorimetric Sensor, Euqlidean Distance, RGB, cyanide ion*.

Abstract

Sensor to detect cyanide ion (CN^-) has been successfully conducted by sol-gel method using tetraethyl orthosilicate precursor and $FeCl_3$ reagent. This study aims to determine the optimal conditions of synthesis, optimal conditions of the sensor, determine LoD and LoQ. The synthesis was carried out by mixing the precursor with ethanol solvent. H_2O, Triton X-100, reagents and catalysts were added to the mixture. The variations carried out were the concentration of reagents and the best aging time. The sensor results are expressed in the Euclidean Distance (ED) value of the Red-Green-Blue (RGB) dots obtained. Optimal conditions for censoring were performed by varying the contact time. In sol gel synthesis, the best reagent concentration was obtained at 0.1 M concentration and the best aging time was 4 days. Determination of optimal sensor conditions occurred at a time of 30 seconds with a washout limit of 120 seconds. Validation of the sensor method resulted in linearity in the concentration range of 100 - 1000 ppm with an R^2 value of 0,9984. LoD and LoQ respectively were 65,45 ppm and 218,16 ppm. Sensor characterization using FTIR spectrophotometer from wave number 4000 to 400 cm^{-1}. The resulting IR spectrum shows success in sol-gel synthesis, because in this study SiO_2 appears at wave numbers around 433 cm^{-1}. After the sensor stage by immersing the sensor in CN^- 0,01 M and CN^- 0,1 M analysis, there is no sign of the appearance of Fe-S groups at wave numbers 4000-400 cm^{-1}, because the absorption area is at 380-311 cm^{-1}.

How to Cite: Rohsaita, A., Thohir, M., Surya Efendi, M., Kusumaning Tiyas, W., & Nursaida, M. (2024). Sintesis Sensor Kolorimetri untuk Deteksi Sianida dengan reagen Besi (III) Klorida Menggunakan Metode Sol Gel dengan Kombinasi Smarthphone. *Hydrogen: Jurnal Kependidikan Kimia, 12*(3), 474-486. doi:https://doi.org/10.33394/hjkk.v12i3.11887

https://doi.org/10.33394/hjkk. v12i3.11887

INTRODUCTION

Human activities are the largest contributor to pollution in Indonesian rivers, at least 60% of pollution is caused by human actions. Starting from household industrial waste, overfishing and destructive fishing, using explosives and poisons that ultimately have an impact on fish habitat in the river (Permana et al., 2021). Other pollution can come from inorganic materials in the form of metals, salts and acids. This material can come from industrial waste which contains hazardous and toxic compounds, one of which is the cyanide compound (Angraini & Falahudin, 2021).

Cyanide is a gaseous, solid or liquid compound. Cyanide can be widely distributed in waters in the form of cyanide ion (CN^-), hydrogen cyanide (HCN), and potassium cyanide (KCN) (Angraini & Falahudin, 2021). Cyanide has highly reactive properties that can have a negative

impact on living things, namely disrupting liver function, breathing and causing bone damage. Cyanide in the life around us can be found in cigarettes, motor vehicle fumes, and foods such as spinach, bamboo, beans, tapioca flour and cassava (Pratama et al., 2012). Cyanide found in waters comes from industrial waste such as the metal plating industry, gold mining which requires electroplating and metal *polishing*.

Analysis for cyanide ions usually utilizes various types of instruments such as UV-Vis spectrophotometer, SSA and Fluorometric Spectroscopy. Based on the research of Sultan, et al. (2015) the content of mercury and cyanide in the Talawaan River Watershed, North Sulawesi by manual method using a HACH DR 5000 spectrophotometer. The cyanide analysis conducted by Astiti & Sugianti (2014) with the Vapor hydrid method using Atomic Absorption Spectrometry (AAS). In research conducted by (Alonso-González et al., 2017) using a spectrophotofluorometric method that can detect cyanide up to a concentration of 0,026 ppm. However, this method has the disadvantage that there is interference by a small amount of foreign fluorescent material in the laboratory.

This proves that the analysis must be done in a laboratory because it requires specialized instruments. But even though using instruments there are still a number of interferences. In addition, the instrument also requires expensive costs and there are also several disadvantages of using instruments in metal ion analysis, namely the need for complicated preparation, technicians are needed and require a lot of energy. This is in contrast to the 12 principles of "Green Chemistry" or commonly called "Sustainable Chemistry" designed by Anastas and Warner in 1998 (de Marco et al., 2019). Therefore, it is necessary to find a solution to minimize energy efficiency to be able to design chemical products with processes that are more environmentally friendly (Sidjabat, 2008). So, a lot of research has emerged that aims to provide alternative analysis for various types of metal ions in the form of chemical sensors.

Optical chemical sensors for metals are usually done colorimetrically due to their simple nature. The colorimetric technique is based on the change in color intensity that occurs in the sample when the reagent is in contact with the target analyte. The color gradation formed indicates the concentration of the analyte (Gilchrist & Nobbs, 2019). In its development, colorimetric techniques are not only based on liquid-liquid reactions, but also on solid-liquid which is easier to operate and requires fewer reagents (Mizuguchi et al., 2008).

The colorimetric method has the advantage of being more effective and efficient to use because it does not require complicated sample preparation, does not require instruments because it can be observed directly by the naked eye with a color change resulting from the interaction between the receptor and the analyte (Suharman & Rahayu, 2020). As in the research (Zhu et al., 2020) that anthraquinone-based colorimetric sensors can be used for CN- detection in solutions that have good selectivity, high sensitivity and experience color changes that can be seen with the naked eye. One of the methods used to make colorimetric sensors is the sol-gel method. Sol-gel is an inorganic synthesis method that has the principle of changing the phase of substances from sol to gel. According to (Chu & Chuang, 2015), this method can accommodate several reagents for different target analytes, so the sol-gel used as a reagent immobilization matrix is quite broad and flexible. In addition, this method is easy to control the results of the synthesis because each stage greatly affects the results of the synthesis.

The ease of controlling the synthesis results in the wide application of this method. The sol-gel method will produce a porous surface as a result of the hydrolysis and condensation of

alkoxide groups in precursors made in an acidic or alkaline atmosphere. The aging process in the sol-gel method carried out at room temperature will make the reagents in the mixture of sol-gel method results entangled in the silica matrix (Avnir et al., 1984). The pore size formed will be adjusted to the needs of each reagent used in the manufacture of sensors, the selection of optimum conditions in the manufacture of silica gel will make the pores not too small and make the reagent activity decrease and not too large which makes the reagent leached easily (Tang et al., 2003). so that the optimization process in making optical chemical sensors using the sol-gel method is a mandatory thing to do.

This research will use Iron (III) reagents that can produce color changes when reacting with cyanide (CN^-). Detection with this reagent has a high sensitivity, which is 1 μm CN^- with a concentration limit of 1 in 50.000 (Lin et al., 2013). In addition, there are other tests used in colorimetric detection of cyanide, namely the copper acetate-benzidine test with a higher sensitivity, namely 0.25 μm CN^- with a concentration limit of 1 in 200.000, but the reagent is carcinogenic (Lin et al., 2013) In addition, in making a detector that is really simple and can be operated easily, this study will use the red-green-blue (RGB) value of the color formed in quantifying the analysis results (Feng et al., 2011). The RGB price is converted into a value that can distinguish each color gradation with the Euclidean Distance (ED) equation. In this study, a smartphone camera was used for the quantification process (Leonard et al., 2022). The Euclidean Distance (ED) approach is able to complement the shortcomings of colorimetric sensors by simplifying the quantification process without worrying about defining colors, which according to each researcher tends to be subjective (Effriandi et al., 2019). In addition, this sensor can also be an alternative detector for cyanide metal ions which is not only easy to manufacture but also easy to operate.

RESEARCH METHODS

Tools and Materials

The tools used in this research are petri dishes, beaker glass, hotplate stirrer, micropipette, measuring pipette, magnetic rod, black box, smartphone, tweezers, glass stirrer, analytical balance and whatman 42 filter paper. The materials used were iron (III) chloride ($FeCl_3$), aquabides (H_2O), Triton X-100 ($C_{14}H_{22}O(C_2H_4O)n$), tetraethylorthosilicate ($Si(OC_2H_5)_4$), ethanol (C_2H_6O) and hydrochloric acid (HCl).

Sol-gel synthesis of sensors

Determination of the best reagent concentration

The first thing that was done was to mix TEOS with ethanol, 2 mL each, with stirring for 30 minutes, carried out in 4 beakers. Then to the solution was added 683 μL of aquabides, 10 drops of Triton X-100, $FeCl_3$ solution with concentrations of 0,01 M; 0,05 M; 0,1 M; and 1 M as much as 2 mL, and HCl 0,3 M as much as 0,5 mL which was added drop by drop. All materials were stirred for 5 hours until homogeneous. Immersion of Whatman 42 filter paper that has been prepared by cutting the size of 1 × 1 cm into the sol solution with different composition of $FeCl_3$ solution and stored in a light-proof room for 24 hours. Then the filter paper is drained in a cup and dried by airing it to form a wet gel. filter paper is oven first at 40°C before use, so that the wet gel can become xerogel and filter paper can be used as a cyanide sensor.

Cyanide analytes with concentrations of 0,01 M and 0,1 M were prepared and then filter paper was dipped into the sensor for 3 minutes to form a color. The sensor that has formed the color is photographed using a smartphone with the help of a black box. From the color obtained, the RGB value is then sought and converted into the Euclidean Distance (ED) value.

Determination of the Best Aging Time

The best reagent concentration was done by differentiating the reagent concentration in the sol mixture. First mix TEOS with ethanol, 2 mL each, with stirring for 30 minutes, done in 4 beakers. Then to the solution was added 683 µL of aquabides, 10 drops of Triton X-100, $FeCl_3$ solution with a concentration of 0,01 M; 0,05 M; 0,1 M; and 1 M as much as 2 mL, and HCl 0,3 M as much as 0,5 mL which was added in a drop-by-drop manner All materials were stirred for 5 hours until homogeneous. Then immersion was carried out on Whatman 42 filter paper that had been prepared by cutting the size of 1 × 1 cm into the sol solution with the composition of different $FeCl_3$ solutions and stored in a light-proof room for 24 hours. Then the filter paper is drained in a cup and dried by airing it to form a wet gel. filter paper is oven first at 40°C before use, so that the wet gel can become xerogel and filter paper can be used as a cyanide sensor.

Analytes with concentrations of 0,01 M and 0,1 M were prepared and then filter paper was dipped into the sensor for 3 minutes to form a color. The sensor that has formed the color is photographed using a smartphone with the help of a black box. From the color obtained, the RGB value is then sought and converted into the Euclidean Distance (ED) value. Filter paper that has been coated by xerogel is measured every 2 days from day 2 to day 14, using different paper.

Performance Testing

Determination of Optimum Time for Censorship

Cyanide solutions of 0,01 M and 0.1 M concentration were put into 10 bottles. The sensors were contacted with time variations of 3, 6, 9, 12, 15, 18, 21, 24, 27, and 30 seconds. Each variation was contacted with 2 paper sensors. Then the sensor is photographed at 0, 30, 60, 90, and 120 seconds using a smartphone with the help of a black box. Then the RGB value, ED value and significance of variance analysis were determined.

Leaching Test

In the leaching test there are 2 treatments without using aquabides and with aquabides. First by using aquabides, 10 bottles filled with aquabides were prepared. The sensor was dipped for 30, 60, 90, 120, 150, 180, 210, 230, 250, 270, and 300 seconds into aquabides. Then aerated to dry. After that, it was dipped in cyanide solution with a concentration of 0,01 M and 0,1 M until a color change occurred. The color formed was then photographed using a smartphone with the help of a black box and determined the RGB value, and converted to ED value.

For treatment without using aquabides, the sensor was first dipped for 30, 60, 90, 120, 150, 180, 210, 230, 250, 270, and 300 seconds into cyanide solutions with concentrations of 0,01 M and 0,1 M until color changes occurred. The color formed is then photographed using a smartphone with the help of a black box and the RGB value is determined, and converted into an ED value.

Determination of Validation Parameters

Determination of Linearity

10 bottles were prepared and filled with cyanide solutions of different concentrations. The concentrations used were 100, 200, 300, 400, 500, 600, 700, 800, 900 and 1000 ppm. contacted for 30 seconds. The sensor that has formed the color is photographed using a smartphone with the help of a black box. From the color obtained, the RGB value is then sought and converted into the Euclidean Distance (ED) value.

Detection Limit and Quantification Limit

10 sensors were prepared as blanks. The ten sensors that have formed the color are photographed using a smartphone with the help of a black box. From the color obtained, the RGB value is then sought and converted into the Euclidean Distance (ED) value. From the ED value obtained, the standard deviation is calculated with Ms. Excel. Then, the standard deviation value obtained is entered into the following equation.

$$\text{LoD} = \frac{3 \text{ x Sb}}{\text{m}}, \text{ LoQ} = \frac{10 \text{ x Sb}}{\text{m}}$$

Characterization of Sensors

The synthesized sensor was then characterized using an FTIR spectrophotometer with wave numbers 4000 cm^{-1} to 400 cm^{-1}. The materials characterized were aquabidest, Whatman 42 filter paper, wet gel after synthesis, dry gel, iron (III) chloride, filter paper coated with wet gel before sensing (Fe 0,01 M and 0,1 M), filter paper coated with wet gel after sensing and tetraethyl orthosilicate/TEOS.

RESULTS AND DISCUSSION

Sol-gel synthesis of sensors

Determination of the best reagent concentration

Determining the best concentration is related to determining the maximum capacity of the matrix to encapsulate reagents. If the reagent is too little, it can reduce sensitivity while too much reagent can occur leaching. The purpose of sensor analysis is to cut the use of detection reagents but also to detect with a high level of sensitivity, so that the determination of the optimum concentration can be used to determine the limit between the lowest sensitivity point of the analyte that can be detected with the leaching that occurs. This experiment uses various concentration variations ranging from concentrations of 0,01 M, 0,05 M, 0,1 M and 1 M. The results of the research at this stage can be seen in Figure 1.

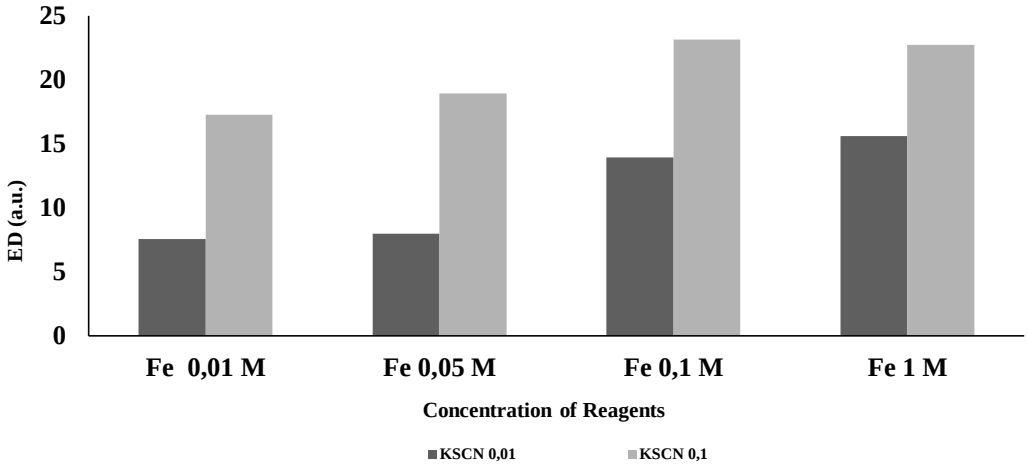

Figure 1. Graph of the effect of reagent concentration on ED values

Figure 1 shows that the increase in concentration greatly affects the increase in the ED value in each sensor. Of the three sensors with different reagent concentrations, namely Fe 0,01 M, 0,05 M and 0,1 M did not cause visible leaching in the residual solution of the analytes used. The results of determining the best reagent concentration showed that up to a concentration of 0,1 M, the sol gel matrix was able to encapsulate the reagents well. In addition, testing was also carried out at a higher concentration, namely at a concentration of 1 M, but the reagent at a concentration of 1 M had experienced visible leaching. Therefore, a concentration of 0,1 M was taken as a suitable concentration for the sol-gel matrix made because it has a high ED value and is also free of visible leaching.

Determination of the Best Aging Time

Aging time is something that will affect the structural character and mechanical strength of the synthesis results. Meanwhile, the aging process is a stage that can separate the sol form into a wet gel. At this stage there will generally be changes in the silica structure from Si-OH from hydrolysis to Si-O-Si. This stage will affect the length of time required in the aging stage. sol-gel applied to sensors takes up to 14 days, but the required time can be obtained in less than 14 days. Therefore, in this study, the sensor performance was measured from day 2 to 14 to find out how long the aging process required by the synthesis results when the supporting media was Whatman 42 filter paper.

Figure 2 shows that there is a sharp increase in intensity immediately on day 2. Although there is still an increase as it goes to day 4, and starts to remain constant from day 4 to day 14. From this data, it can be concluded that with filter paper media, the sol-gel has achieved a good aging time even though it is only 2 days. This is very different from sol-gels bonded to glass media as reported in (Samadi-Maybodi et al., 2015) and (Shahamirifard et al., 2018) which can require up to 14 days of aging.

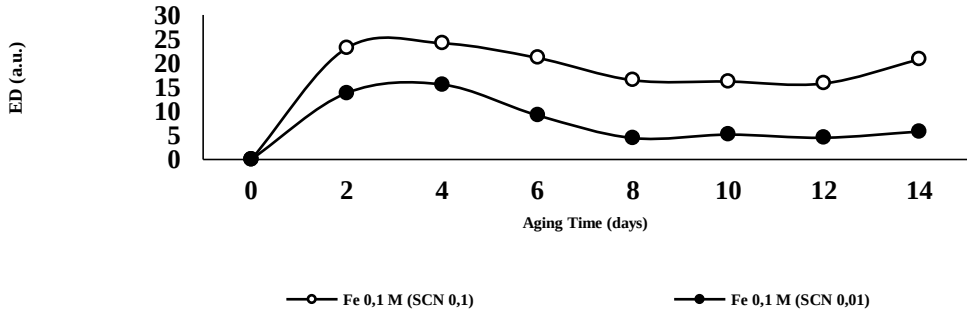

Figure 2. Curve of the effect of aging time on ED value

Silica sol can fill the pores of the filter paper, which is very different from the glass media that utilizes the interaction between the glass surface filled with silanol groups. It is expected that there will be interaction between the glass surface and silica sol. In addition, the pores of glass are also much smaller than paper when physical interactions are formed. In the paper matrix, the gel that has formed Si-O-Si fills the cavities of the filter paper without forming chemical bonds or other interactions with the surface of the support medium. However, from the graph above, day 4 was chosen as the optimum aging time because day 4 is the maximum value that the sensor can achieve in the censorship stage. After the 4th day, the ED value is relatively constant which indicates that the aging process of silica gel on the paper matrix by forming a structure that suits the needs of the sensor has been completed.

Performance Testing

Determination of Optimum Time for Censorship

Contact time is the time required by the sensor in the censoring stage with a marked change in color as a result of the reaction between the analyte and the reagent. Contact time is an important factor in sensor analysis.

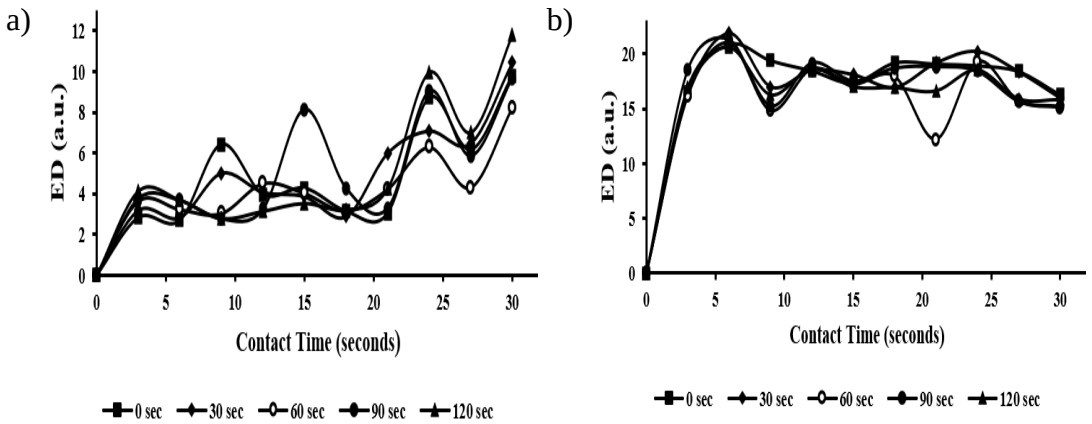

Figure 3. (a) Curve of the effect of sensor contact time on ED value with CN^- 0,01 M concentration; (b) Curve of the effect of sensor contact time on ED value with CN^- 0,1 M concentration.

Figure 3(a) shows that the sensor can detect CN^- optimally with a low concentration if it is in contact with the sensor for 30 seconds. This is different from Figure 3(b) where the sensor is able to achieve optimum performance in only 5 seconds of contact time. The faster contact time is because the sensor used uses colorimetric principles that are simpler and easier to operate.

Leaching Test

Leaching tests are among the most important stages carried out to determine the potential of reagents that can be released from the sensor and the effectiveness of reagents on the sensor to interact/mix with the surrounding liquid (Shahamirifard et al., 2018). In this leaching test, two stages were carried out, namely the first immersion with aquabides and without immersion with aquabides directly with the CN^- analyte. In sensors that have been immersed in aquabides before, it may no longer be able to detect CN^- with better accuracy because there may be some analytes that are released from the sensor side.

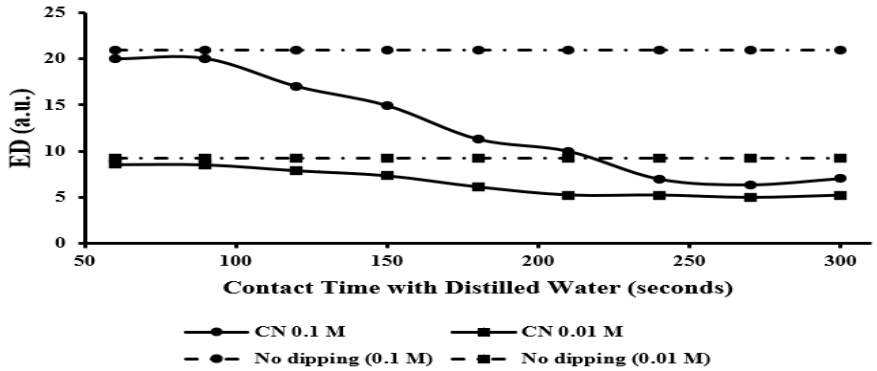

Figure 4. Curve of the effect of sensor contact time with aquabides and without aquabides on ED value

Figure 4 shows that there is a difference in ED value between contacted with aquabides and without immersion. In the figure, the ED value of the sensor without dipping with aquabides at CN^- 0,01 M immersion is 9,27 and at CN^- 0,1 M immersion is 20,93. The results obtained from this leaching test are that when contacted with aquabides for 120 seconds at all concentrations of both CN^- 0,01 M and CN^- 0,1 M that the sensor has begun to leach significantly. So it can be interpreted that if the time in immersing the sensor in the analyte is more than 120 seconds, there will be a decay of the reagent on the sensor. Thus, the ability of the sensor to detect reagents will not take place to the maximum.

Determination of Validation Parameters

Determination of Linearity

Validation of the method aims to test the correctness of the values obtained during the research. Validation of the method is carried out at optimum conditions in accordance with those

obtained during the process of determining the optimum time. Method validation includes determination of linearity, limit of detection (LoD) and limit of quantification (LoQ). Linearity test was performed by comparing the difference in concentration with the ED value with the presence of color pegs. Determination of the linear region by showing where the concentration region is located which gives a constant increasing response to the ED value obtained. This linearity test was carried out with a concentration range of 100 to 1000 ppm. The linearity data will show the y equation, and the equation will also know the slope value which states the sensitivity of the sensor made, besides that the R^2 value will also be known. The linearity data is shown in Figure 4.

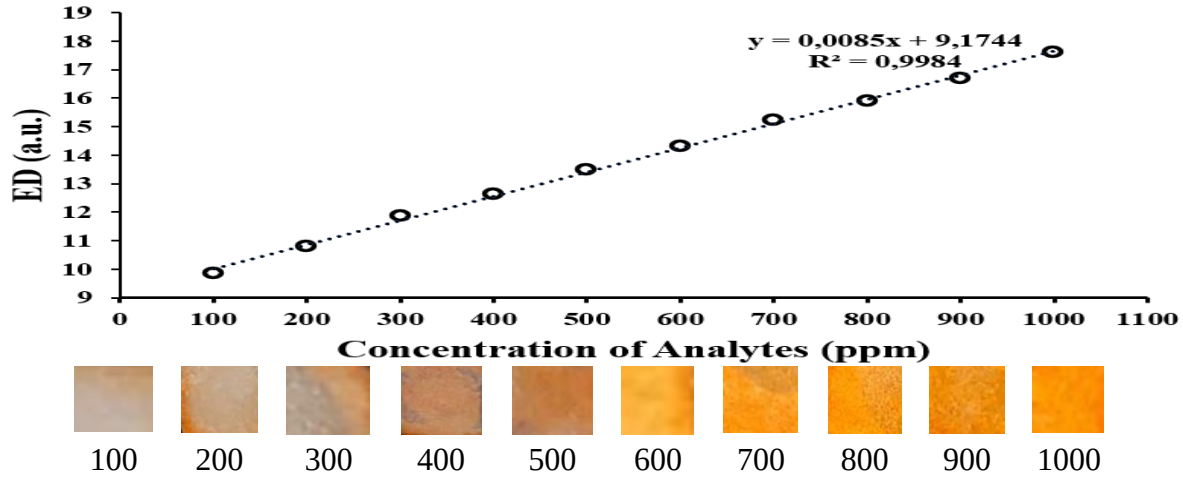

Figure 5. Sensor Standard Curve

Detection Limit and Quantification Limit

The limits of detection and quantification were calculated based on the standard deviation of 10 sensors in the linearity test. The results obtained for the limit of detection (LoD) were 65,45 ppm and the limit of quantification (LoQ) was 218,16 ppm. The smaller detection limit indicates that the sensitivity value of the new method is made. While the limit of quantification shows the smallest quantity that can give accurate results. From the results obtained, it can be concluded that the sensitivity and lowest detection level of the sensor is good.

Characterization of Sensors

Sol-gel synthesis of sensors

The material contained in the gel sol synthesis process is characterized using an FTIR spectrophotometer to determine the functional groups possessed. In this gel sol synthesis, it can be said to be successful if there is absorption that appears in a specific region that shows the absorption of functional groups from the synthesis results. there are main ingredients in the synthesis of gel sol, namely TEOS and water, Iron (III) Chloride reagents are also involved in the synthesis process and will eventually produce SiO_2 networks, so that the absorption of functional groups that should be in this characterization is the -OH functional group from water,

Si-O or O-C from TEOS, and Si-O-Si from the synthesized xerogel. The characterization results are shown in Figure 6.

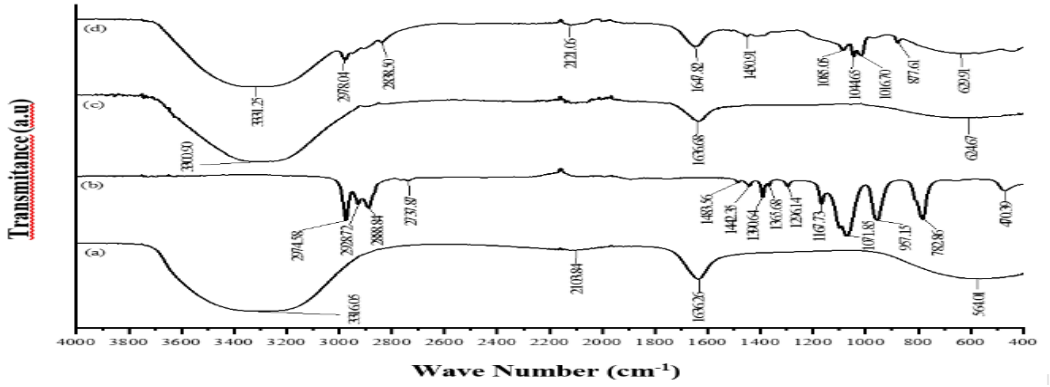

Figure 6. IR spectra of synthesis stage (a) Aquabides (H_2O), (b) TEOS, (c) $FeCl_3$ 0.1M and (d) Wet Gels

In Figure 6(a) is the IR spectra for H_2O, in this spectra there is an absorption in the area around 3316,05 cm^{-1} that refers to a typical peak for the stretching vibration of the -OH group (hydroxyl group). In addition, there is one other absorption at 1636,26 cm^{-1}, which is the bending vibration of the -OH group of water molecules (Simatupang & Devi, 2016). Furthermore, Figure 6(b) is the IR spectra for TEOS, in this spectra an absorption band appears at the wave number 470,39 cm^{-1} showing the bending vibration of the siloxane group (Si-O-Si). In addition, an absorption band appears at wave number 782,86 cm^{-1} which shows the Si-O stretching vibration of siloxane (Triviana et al., 2015). The Si-O stretching vibration on silanol (Si-OH) is shown by the absorption at wave number 957,15 cm^{-1}. The strong absorption band at wave number 1077,85 cm^{-1} is the symmetrical Si-O stretching vibration of siloxane (Si-O-Si) (Triviana et al., 2015). Figure 6(c) is the spectra for $FeCl_3$ reagent with a concentration of 0.1 M, in the wave number range of 4000-400 cm^{-1} there is no absorption for Fe-Cl, because according to (Böhm et al., 2015), $FeCl_3$ is in the absorption region between 98-248 cm^{-1}. This causes the spectra of $FeCl_3$ on the FTIR graph to resemble Aquabides (H_2O). Figure 6(d) is the IR spectra for the wet gels from the synthesis. In the spectra of the wet gel there is absorption at wave number 1083,86 cm^{-1} which is the absorption of Si-O, there is a peak at 877,58 cm^{-1} which is the Si-O-Si stretching vibration (Simatupang & Devi, 2016). In the wet gel phase there are still many compounds of the initial ingredients of the synthesis, because all of them have not evaporated. This can be seen from the absorption of 1647,72 cm^{-1} and 3331,32 cm^{-1} appearing from O-H silanol bending vibrations (Wulandari et al., 2019). The bands that appear in the spectra of this wet gel indicate that the functional groups contained in the wet gel result from the hydrolysis process and the condensation process, namely silanol groups (Si-OH) and siloxane groups (Si-O-Si).

The Censorship Stage

In the staining stage, not only tracking the absorption that will arise from the interaction between CN^- and the reagent, but also the interaction between the xerogel with the supporting solid material and the interaction of the reagent that has been immobilized in the sol-gel matrix. The results of the characterization of filter paper, xerogel coated filter paper before censoring, after censoring with CN^- 0,01 M and after censoring with CN^- 0,1 M can be seen in Figure 7.

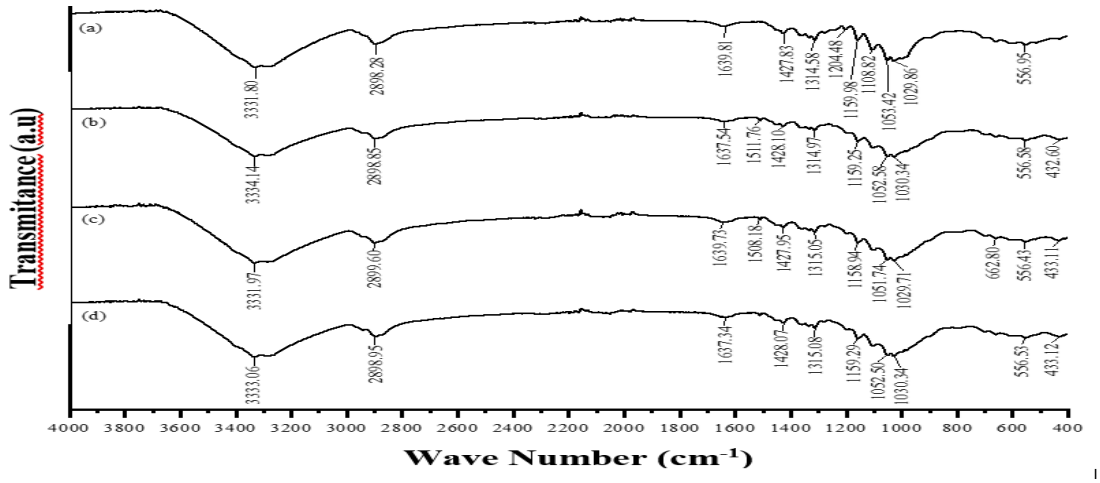

Figure 7. IR spectra of the censorship stage (a) Blank filter paper, (b) xerogel coated filter paper, (c) after censoring with SCN 0,01 and (d) after censoring with SCN 0,1.

In Figure 7(a) is the IR spectra for Whatman 42 filter paper. There is an absorption at 3331,80 cm^{-1} which refers to the vibration of the -OH group. The absorption at 1029,86 cm^{-1} is the stretching vibration of C-O-C. Whatman 42 filter paper contains -OH and C-O-C functional groups because there is a composition of paper forming cellulose which has -OH and C-O-C groups found in the cyclic and connecting parts between monomers (Hua et al., 2010). Meanwhile, Figure 7(b) is the IR spectra for xerogel coated filter paper. There are absorptions at 1052,58 cm^{-1}, 556,58 cm^{-1} and 432,60 cm^{-1} respectively which are contributions from Si-O, siloxane groups and Si-O-Si stretching. Figures 7(c) and 7(d) are IR spectra at the stage after censoring with different concentrations of SCN. In this spectra, there are absorptions at wave numbers 1639,73 cm^{-1} and 1637,64 cm^{-1} which are bending vibrations of O-H-O. However, after the censorship stage by immersing the sensor in CN^- 0.01 M and CN^- 0.1 M analytes, there is no sign of the appearance of Fe-S groups at wave numbers 4000-400 cm^{-1}, because the absorption area is at 380-311 cm^{-1}(Guo et al., 2017). In addition, the absorption of wave numbers around 3300 cm^{-1} shows the nature of the sol-gel matrix which is very hydrophilic because there are many water compounds in the sol-gel matrix (Stanley & Nesaraj, 2014).

CONCLUSION

In this study, the synthesis of sol gel for sensors obtained the best reagent concentration at $FeCl_3$ 0,1 M concentration and the best aging time for 4 days. The optimum condition for censoring occurs at a contact time of 30 seconds and this condition has a washout limit of 120 seconds. The LoD and LoQ values of the linear standard curve in the concentration range of 100 - 1000 ppm respectively were 64,53 ppm and 218,42 ppm.

SUGGESTION

There is a need for variation during the synthesis process because there is still much that can be modified such as the manufacturing method, type of reagent and other types of supporting materials.

ACKNOWLEDGMENT

The author would like to thank the supervisors for their extraordinary guidance and support so that I can carry out this research well. The author would also like to thank all those who have contributed to this research.

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