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Evaluation of the Adsorption and Reduction of Au(III) on Esterified Humic Acid

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Abstract-Electronic waste is increasing every year, one of which is electronic waste metal. Gold mining is less profitable than taking gold from electronics. One of the adsorbents that can be used is humic acid. To study the effect of functional groups on the adsorption of Au (III) ions, the adsorptionreduction of Au (III) ions using esterified humic acid was studied. Humic acid that will be esterified is isolated from peat soil from Rawa Pening, Central Java. Esterification was performed by reaction with ethanol in acidic conditions and doing repeated. Estesterified humic acid was characterized by Fourier-Transform Infrared spectroscopy (FTIR), X-Ray Diffraction (XRD), ash content, total acidity, -COOH content, and -OH content, then used for adsorption-reduction of Au (III). Esterified humic acid has studied the effect of pH adsorption, adsorption kinetics, and adsorption isotherm on adsorption reduction process of Au (III). The Au metal formed was confirmed by XRD diffractogram and photo-optical microscope. Esterified humic acid has 0.78% ash content, total acidity of 1022 cmol/kg, COOH content of 217 cmol/kg, and -OH content of 805 cmol/kg. pH optimum on adsorption reduction Au(III) using esterified humic acid was 6. The adsorption capacity is 625 mg/g with Langmuir isotherm model esterified humic acid. The adsorption rate constant (k) is 1.50.10-3 g/mg min with Ho kinetic model for esterified humic acid. Peaks of gold particles in the diffractogram are detected on $2\theta = 38$, 44, and 64.

Keywords—adsorption, reduction, Au (III), humic acid, esterification

I. INTRODUCTION

Electronic such as televisions, radios, smartphones, and computers contain gold because they can conduct electricity. Yokohama Metal Co. Ltd. (Japanese company) did research and found that the content of 1 ton of gold mine material after processing on average only produced 5 grams of gold, while 1 ton of used cellphones could produce 150 grams of gold or more. The handphone recycling industry in Japan, Eco-system Recycling Co., produces gold bars between 199.58 to 299.37 kg/month. Mining gold used cellphones produces gold that is almost the same as a small scale gold mining, with less risk and capital [1].

Gold (Au) is a heavy metal that has high economic value, so it is interesting to explore [2]. Gold in nature is found in the form of metal (native) found in cracks of quartz rock and the form of minerals. Gold is also found in the form of alluvial gold, which is formed from the weathering process of rocks containing gold. Gold in the earth's crust is estimated at 0.004 g/ton, including around 0.001 g /ton found in marine waters[3]. Gold is widely used as jewelry and also has many benefits in the health sector, such as for beauty, composing nanoparticles in cancer treatment, used in the pharmaceutical industry, and others.

The method for obtaining the most used gold metal is the extraction method using cyanide [4] and the amalgamation method using mercury [5]. Cyanide is used as a gold extracting material for more than 100 years because it is cheap [2]. Mercury and cyanide can enter and settle in the waters and enter into the food chain when it comes to the human body through water that is used for daily needs and the water products that humans use. This results in the accumulation of heavy metals in the body's tissues so that it can slowly cause permanent damage to the body's organs and most severely can cause death [1].

The method that can be used to produce gold without mercury and cyanide is adsorption. The adsorption method is widely used because it is cheap, easy to use, simple, and has the capacity to produce large metals [7]. Adsorption is carried out based on the interaction between metal ions and functional groups on the surface of the adsorbent through complex formation interactions. The interactions of metal ions and functional groups usually occur on the surface of solids which are rich in functional groups such as –OH, -NH, -SH, and – COOH [6].

The adsorbents in gold hydrometallurgical processes are widely used, among others, are activated carbon [8], chitosan resin[9], persimmon tannin gel, novel tanning gel [10], lignofenol derivatives [11], phenol derivatives [12], diethylaminoethyl-cellulose [13], humin [14], humic acid [1].

Hamamoto et al [12] have reduced gold ions to gold metals using phenol derivatives and showed the results that the hydroxyl groups in phenol derivatives work efficiently in reducing gold ions to gold metal.

Humic acid is one of the fractions of peat soil that can dissolve in alkaline but is not soluble in acid. Humic acid contains functional groups –COOH and -OH so that it can be used as an adsorbent for metals and organic compounds [15].

Esterification can be used to determine whether there is an influence of -COOH in humic compounds to interact with metal ions. References [16] has conducted research on esterification of peat soil, humic acid, and humin and its effect



on the binding of Cu (II) ions and showed that esterification could reduce 80% binding of Cu (II) ions to humic acid.

References [16] also esterified biomass alfalfa then applied it for adsorption of Au (III) ions and showed that the esterification of the carboxylic group was able to reduce the 30.9% Au (III) ion binding capacity. Esterification can be done using several solvents such as methanol [17] or ethanol [12]. The success of esterification of humic compounds is known from the interpretation of functional groups in IR spectra, showed the reduction in the peak intensity of the hydroxyl groups in the area of 3400 cm -1 and the increase in CO peak intensity in the area of 1100 cm -1 as a result of the formation of esters [16].

The amount of electronic waste produced and given the negative impact caused, in this study, we will study the effect of esterification of humic acid on the reduction of Au (III) ion adsorption.

II. METHODS

A. Material

Humic acid that used in this research is isolated from peat soil from Rawa Pening, Central Java. Material chemicals used in research this is ethanol, H2SO4, methanol, chloroform, HAuCl4 solution 1000 ppm, aquades, Whatman 42, and N2 gasses.

B. Instrumentations

The instruments used in this study are hot plate and stirrer, centrifuge (Kokusan type H-107), pH-meter, oven, Fourier Transform Infrared Spectrophotometers, optical microscope, X-ray diffractometers (Shimadzu XRD-6000), and Atomic Absorption Spectrophotometers (Perkin Elmer 3110).

C. Esterification

One gram of pure humic acid added 50 mL ethanol, and 4 mL H2SO4 then refluxed at 65 o C for 4 hours. The filtrate is then extracting with chloroform: water 1:1. The chloroform fraction is filtered and dried. The esterification process is repeated twice. The esterification results measured the ash content and calculated the functional group content and characterized by FTIR, XRD.

D. Determination of Ash Content

Fifty milligrams of esterified humic acid was included in a porcelain dish and then heated in a furnace at 750 o C for 4 hours. The sample weight before and after heating is measured.

E. Determination of Functional Groups

Esterified humic acid soaked 0.1 M HCl overnight, then filtered and dried. The sample was determined by the functional group content, which included the content of the carboxylic group (-COOH) and phenolics (-OH) carried out quantitatively.

Determination of total acidity content: one hundred milligrams of identified esterification humic acid was put into Erlenmeyer then 20 mL of 0.1 M Ba(OH) 2 solution and nitrogen gasses was added. Also prepared a blank solution without the addition of esterified humic acid. Blank and sample solutions are then shaken out 24 hours. The formed suspension is filtered, then the residue is rinsed with 20 mL

of distilled water (CO2 free). The filtrate and rinse water were mixed and then titrated using a 0.5 M HCl solution to pH 8.4.

Determination of carboxylic group content: one hundred milligrams of identified esterification humic acid was added to the Erlenmeyer, 10 mL of a solution of 0.5 M Ca(CH3COO) 2 and 40 mL of distilled water were added (free of CO2). A blank solution without the addition of unified humic acid is also prepared. Blank solutions and samples were shaken out 24 hours. The formed suspension is filtered, and the residue is rinsed with 10 mL of CO 2 free distilled water. The filtrate and rinse water are mixed and titrated with 0.1 M NaOH solution to reach pH 9.8.

Determination of phenolic -OH group content: The phenolic -OH group content was obtained from the difference between the total acidity content and the -COOH group content.

F. Determination of Optimum PH Adsorption

Ten milligrams of esterified humic acid is mixed with 10 ml of AuCl4- a solution with the same concentration and given a pH of 1, 2, 3, 4, 5, 6, 7, and 8. The mixture is then shaken for 24 hours then filtered with Whatman 42 filter paper. The filtrate is then analyzed by an atomic absorption spectrophotometer (AAS).

G. Determination of Adsorption Rate and Adsorption Equilibrium Constant

Ten milligrams of esterified humic acid mixed with 10 ml of AuCl4- a solution with the same concentration at optimum pH. The mixture is then shaken with a time varied from 0, 5, 10, 20, 30, 60, 90, 120,150, 180, 210, 240, 270, 300, 600, 900, 1200, 1500 and 1800 minutes. The mixture was then filtered with Whatman 42 filter paper. The filtrate was then analyzed by an atomic absorption spectrophotometer (AAS).

H. Determination of Au (III) Ion Isotherm Model

Ten milligrams of esterified humic acid mixed with 10 ml of AuCl 4- a solution with concentration varied 5, 10, 15, 30, 45, 60, 80, 100, 150, 200, 300, and 400 ppm at optimum pH. Mix then shaken at the optimum time. The mixture was then filtered with Whatman 42 filter paper. The filtrate was then analyzed by an atomic absorption spectrophotometer (AAS).

III. RESULTS AND DISCUSSION

The main functional group –COOH in humic compounds is known to be responsible for metal ion adsorption capacity [18]. To prove this, the -COOH group in humic acid is closed by esterification. Esterification will be selective towards the -COOH group alone without affecting other functional groups. The esterification reaction on humic acid is carried out by reacting between humic acid and ethanol in acidic conditions.

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Fig. 1. IR spectra: (a) humic acid (Prasasti, 2012); (b) 1 times esterified humic acid; (c) 2 times esterified humic acid

Figure 1 shows the difference in IR spectra between humic acid obtained by Prasasti, 2012 compared to one-time esterified humic acid, and two times esterified humic acid. Humic acid after esterification processes, the wavenumber shifts from 1712 cm⁻¹ to wave number 1720 cm⁻¹. This indicates that esters have begun to form. Wavenumber 1712 cm⁻¹ is absorption from the group C = O of –COOH and experiences a shift towards the wave number at 1720 cm⁻¹ where it is absorption from the C = O group that comes from –COOR. In the first esterification, there was no shifting in wavenumber 3371 cm⁻¹, which showed that there was no change in the -OH group and significant intramolecular hydrogen bonds. After esterification two times, wave number 3371 cm⁻¹ shift to wave number 3410 cm⁻¹ and wave number 1720 cm⁻¹ does not experience a shift. This shows that after esterification is two times, the hydrogen bond decreases, supported by the sharper peak of 3410 cm⁻¹. In addition, after two times esterification has been emitted, a CO peak appears in the area of 1100 cm⁻¹, with a peak at the wave number 1095 cm⁻¹ as a result of the formation of methyl esters. This is in accordance with Reference [16] which states that the success of the esterification process can be seen from the reduced intensity of the peak of the hydroxyl group in the area of 3400 cm⁻¹ and the increase in the intensity of the CO peak in the area of 1100 cm⁻¹ as a result of the formation of esters.



Fig. 2. Diffraction of esterified humic acid

Esterified humic acid is then determined by ash content and functional group content. Humic acid ash content after esterification of 0.78%. The characterization of functional groups in esterified humic acid was carried out as in isolation humic acid, which included total acidity, functional group content –COOH, and total functional group content –OH. The total acidity of the esterified humic acid was determined by the Ba(OH)2 method using indirect potentiometric titration. The -COOH group content of esterified humic acid was determined by the Ca-acetate method. The functional group content of -OH total is obtained by calculating the difference between total acidity and functional group content –COOH. Humic acid after esterification, the -COOH group has decreased. This proves that the esterification reaction has taken place even though not all of the -COOH groups have been staged. The -OH group after esterification has increased.

It is possible that before the esterification process, the -COOH and -OH groups in humic acid bond with each other hydrogen.

In the esterification process, namely during the heating process of hydrogen bonds between the -COOH group and the

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-OH group on humic acid, the breakdown occurs. The -COOH group in humic acid will be staged, and the -OH group is free so that after the esterification, the -COOH group decreases and the -OH group increases in number.

BLE I.	FUNCTIONAL (GROUP CONTENT	OF HUMIC ACID AN	ND ESTERIFIED HUMIC ACID
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	Acidity content (cmol/kg)	-COOH (cmol/kg)	-OH (cmol/kg)
Humic Acid (Prasasti,2012)	799	406	393
Esterified Humic Acid	1022	217	805

A. Optimum PH Measurement

To study the effect of pH on the adsorption-reduction process, a solution of Au (III) pH of the solution was made a variation from pH 1 to pH 8.

At pH 1, percent adsorption-reduction is low. As with adsorption with humic acid, it is possible at pH 1 that the -COOH group and -OH in the esterified humic acid tend to form hydrogen bonds. At pH 2, the adsorption-reduction process increases. The intramolecular hydrogen bond between the -COOH and -OH groups decreases. At pH 2, the –COOH group tends to be positively partial, and Au (III) solution is in the AuCl 4 species - which has a negative partial mutation, so that electrostatic interactions can occur.

At pH> 3, percent adsorption-reduction tends to increase, and from the results of the study indicate that the Au (III) ionreduction adsorption process showed optimum results at pH 6.



Fig. 3. Effect of pH on Au (III) adsorption on esterified humic acid

The number of -COOH groups on esterified humic acid is reduced than pure humic acid, but the -OH group increases, so that it is possible that the reduction process is more dominant than the adsorption process. This is supported by the results of research that shows the optimum results at pH 6. At pH 6, the abundance of H ⁺ in a little solution, so that the -COOH group tends to deprotonate to $-COO^{-}$. At pH 6, the Au (III) species is in the form of AuCl_{4-n}(OH)_n⁻. The reduction potential of AuCl₄⁻ +1 V does not differ greatly from the reduction

potential of AuCl_{4-n}(OH) $_{n}$ which is between +0.7 V to +1 V [19], so that it does not significantly affect the ion reduction process Au (III). As with humic acid, the pH of the solution after the adsorption-reduction process has decreased due to the reduction of the Au (III) ion into Au (0) ions, each mole of reduced Au (III) releases 3 moles of H $^+$ ion in the solution which causes the solution becomes acidic as shown in Table 2.

TABLE II. RESULTS OF PH MEASUREMENTS OF SOLUTIONS BEFORE AND AFTER ADSORPTION

1 1,190 1,186 2 2,229 2,189 3 3,105 2,960 4 4,002 3,339 5 5,108 3,454 6 6,040 3,480 7 7,003 3,541 8 8,000 2,722	er Adsorption	pH Solutions Before Adsorption	pН	
2 2,229 2,189 3 3,105 2,960 4 4,002 3,339 5 5,108 3,454 6 6,040 3,480 7 7,003 3,541 8 8,000 2,722		1,190	1	
3 3,105 2,960 4 4,002 3,339 5 5,108 3,454 6 6,040 3,480 7 7,003 3,541 8 8,000 2,722		2,229	2	
4 4,002 3,339 5 5,108 3,454 6 6,040 3,480 7 7,003 3,541 8 8,000 2,722		3,105	3	
5 5,108 3,454 6 6,040 3,480 7 7,003 3,541 8 8,000 2,722		4,002	4	
6 6,040 3,480 7 7,003 3,541 8 8,000 2,722		5,108	5	
7 7,003 3,541		6,040	6	
9 8 000 2 722		7,003	7	
8 8,000 5,752		8,000	8	



B. Kinetics of Adsorption-Reduction

Adsorption and reduction of Au (III) kinetics in esterified humic acid was studied by interacting between esterified humic acid and Au (III) solution at various times until adsorption equilibrium was obtained. Au (III) ions will interact more and more with the active sites of estrogenic humic acid over time until they reach equilibrium conditions





The results showed that the adsorption equilibrium was reached in minutes to 300 (5 hours). After equilibrium is reached, there is no significant increase or decrease in the adsorption-reduction process.

Determination of adsorption rate constant and reduction of Au (III) in esterified humic acid was determined using the kinetic model Langmuir-Hinshelwood, Santosa [21], and Ho[20].

The Langmuir-Hinshelwood kinetics model is calculated by making linear curve equations between vs , so that we get the slope as k and intercept as K. The Santosa kinetics model is done by making linear curve equations between vs produce slope as k and intercept as K. The Ho second order pseudo adsorption kinetics model is done by making a curve equation vs t, so slope as and intercept as , where h = kq e 2.

Of the three kinetic models, Ho kinetic model, which gives a correlation factor (r), which is better than the Santosa and Langmuir kinetic model. The adsorption-reduction process of Au (III) in esterified humic acid so that it follows Ho's second-order pseudo kinetics model, with the price of the Au (III) adsorption rate constant is 0.0015 g / mg minutes.

Model		Parameter	Nilai	
Langmuir-Hinshelwood		R	0,917	
		k (menit ⁻¹)	0,0043	
		K (L/mol)	-10448	
Santosa		R	0,994	
		k (menit ⁻¹)	0,0087	
		K (L/mol)	3951,4	
Но		R	0,999	
		h (mg/g menit)	0,617	
		k (g/mg menit)	0,0015	
100 80 5 40 20			y = 0,05: R ² = 0	x + 1,6185 0,9989
0	500	1000 t (menit)	1500	2000

TABLE III. Results of Calculation of Adsorption-Reduction Kinetics of Esterified Humic Acid

Fig. 5. Pseudo second-order kinetic graph of Ho esterified humic acid.



C. Capacity of adsorption-reduction

The adsorption-reduction capacity of Au (III) in esterified humic acid was determined using adsorption isotherm. The adsorption isotherm model used Freundlich isotherm and Langmuir isotherm. The isotherm equation, according to Freundlich is used to explain the non-ideal adsorption process on a heterogeneous surface of the adsorbent where heterogeneity is caused by the presence of different functional groups on the surface of the adsorbent. Isotherm Langmuir can explain that the adsorption that occurs is monolayer adsorption (one layer) only. If the active site on the surface of the adsorbent is closed in the presence of an adsorbate, then the adsorption process can be blocked.

TABLE IV. RESULTS OF CALCULATION OF THE ADSORPTION-REDUCTION CAPACITY OF ESTERIFIED HUMIC ACID

Freundlich			Langmuir		
$K_F (mg/g)$	n	R	qmax (mg/g)	K _L (L/mg)	r
69,63	1,942	0,772	625	0,091	0,974

From the data from the results of research and calculations, it can be seen that the Au (III) ion-reduction adsorption process in humic acid was quantified following the Langmuir isotherm model. The active site on the surface of humic acid is considered homogeneous, there is no interaction between the molecular adsorbate (Au (III) ion), and the adsorption of Au (III) in esterified humic acid only occurs on one layer (*monolayer*). The adsorption capacity of esterified humic acid in adsorbing Au (III) reduction was a maximum of 625

mg/g, meaning that 1 gram of esterified humic acid was able to adsorb 625 mg of gold, with a value of 0.091 $K_{\rm L}$

Esterified humic acid compared with the humic acid adsorption capacity of 192 mg/g^[1], the adsorption capacity of humic acid was esterified higher, this is because the -OH group content in esterified humic acid was much greater than that of humic acid. So it can be concluded that the reduction process of ions (Au) III is greater than the Au (III) adsorption process in humic acid.



Fig. 6. Graph of the Langmuir isotherm of adsorption of Au (III) ions in esterified humic acid.

D. Characterization of Esterified Humic Acid After the Adsorption-Reduction Process

Esterified humic acid after the adsorption process was characterized by the microscope, FTIR, and XRD photos. The results of a microscope photo were seen in Au (0) metal in an esterified humic acid. This proves the occurrence of a reduction process in almost all surfaces of esterified humic acid. The IR spectra before and after the adsorption-reduction are shown in Figure 8. After the adsorption-reduction process, the wavenumber 3410 cm -1 shift to wave number 3433 cm - 1 and decreases the intensity at that peak which shows that the –OH functional group decreases because the –OH group is

used in the process of Au (III) reduction. The absence of uptake in functional groups -C = O in -COOH at wavenumbers 1720 cm -1 in esterified humic acid after the interaction indicates that almost all of the -COOH groups contained in esterified humic acid bind metals. The appearance of uptake at wave number 1057 cm -1 and 833 cm -1 in the spectra of esterified humic acid after the adsorption-reduction process, showed the presence of metals bond to esterified humic acid.



Fig. 7. Photograph of an estesterified humic acid microscope (left) and post-adsorption esterified humic acid (right).





Fig. 8. IR spectra of esterified humic acid: (a) before interacting with Au (III) ions; (b) after interacting with Au (III) ions.



Fig. 9. Diffractogram of esterified humic acid: (a) before interacting with Au (III) ions; (b) after interacting with Au (III) ions.

The results of the X-ray diffractogram shown in Figure 9 show a very clear difference between esterified humic acid before and after the adsorption-reduction process. The appearance of characteristic peaks of gold metal on humic acid was esterified after adsorption in regions 2 thetas 38, 44, and 64 in X-ray diffractogram indicating the presence of Au (0) metal. The three peak characteristics are the same as the peak pattern of the golden diffractogram produced by Reference [10] and[11]). The three peaks of these characteristics indicate that the reflection of the plane [111], [200], and [220] is a face-centered cubic structure with a = 4.06 Å. The presence of Au (0) in esterified humic acid after adsorption showed that Au (III) ions were not only adsorbed but also reduced to Au (0) metal.

IV. CONCLUSION

Estesterified humic acid has 0.78% ash content, total acidity of 1022 cmol / kg, the total content of -COOH group is 217 cmol / kg, and the -OH group content is a total of 805 cmol / kg. The optimum pH condition at pH 6. The results of the calculation of the adsorption rate constant (k) follow the Ho kinetics model is 1.50.10 -3 g/mg minutes. The results of the calculation of adsorption capacity following the Langmuir isotherm model are 625 mg/g. The formation of gold metal is shown by the appearance of peaks in the XRD diffractogram at $2 \square = 38, 44, and 64$

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