Recovery of gold by activated carbon from ammoniacal thiosulphate solution

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ABSTRACT— A novel study has been done to investigate the recovery of gold by using activated carbon from ammoniacal thiosulphate solution. For recovery of gold, 0.1 gm ore was taken and added 100 ml thiosulfate solution of 5% concentration in a pyrex beaker was placed on a magnetic stirrer for 120 minutes at 150 degrees temperature. The next step is to recover gold from the thiosulphate solution. 0.3 g prepared column of activated carbon is used for 1 hour. The recovery method will be adsorption and gold get recovered on the surface of activated carbon. This dissolution is used for AA (atomic absorption) analysis. The absorption of standards and ore solutions are checked by the AA (atomic absorption). The graph of absorption vs concentration in ppm is obtained. The unknown concentration of gold before adding activated carbon is known and represented by C_0. Then a prepared column of activated carbon is dipped in the above solution of concentration C_0. Then gold from the solution gets adsorbed over the surface of activated carbon. Now, the concentration of gold C_t (after adding activated carbon) in that solution needs to be known. Place this solution in AAs (atomic absorption spectrometry), then concentration after activated carbon is known. Now it will be less than concentration C_0. Gold recovery will be 33.33 %. Only 33.33 % of gold is recovered. Different factors that influence the % recovery of gold have been studied.

Index Terms— Complex, Gold, Hydrometallurgy, Recovery, Thiosulphate

1. INTRODUCTION

Gold has been recognized as a prehistoric condition and has become one of the first metals to be mined, mainly because it is found as notes or as rubble within streams. That becomes a call for that with the help of 2000 BC the Egyptians began to mine gold. The demise mask of Tutankhamun has a hundred iron ore. The king monuments of old Ur (modern-day Iraq), have a high percentage of gold. Gold extraction processes start in the kingdom of Lydia by using, a mixture of gold and silver. Gold coins of 24 carats are developed in the time of King Croesus, whose government ruled out from 561-547 BC. (Hudson et al., 2009). It is a soft yellow metal. It does not react with chemicals, although it does well with Regia aqua. We mostly used gold in different fields like catalysis, nanotechnology, and electroplating with excellent properties such as chemical inertness, and ductility. Different extraction techniques are used but for the reason that the cyanidation process in 1887 is used to extract gold. As a result of its use, animals and humans are at stake. (Crespo, Gimeno, Laguna, Kulcsar, & Silvestru, 2009)

The mining of gold destroys by attracting natural scenes infecting the source of water and leading to important degradation. Other toxic substances are introduced into our surroundings as a result of poor gold mining. Modern commercial gold mines pollute the environment and create toxic waste. Due to the usage of polluting species have open holes in mines and cyanide can keep those mining companies from producing 20 tons of radioactive waste containing a 0.333-ounce gold ring as a result of the leaching process. (Gock, Cordova Equivar, & Review, 1995). Different metals, such as zinc, arsenic, antimony, and nickel, may affect the return of gold and silver to ores in the traditional cyanide processes. Leaks with thiosulfate reduce disturbance to these external surfaces. Cyanide can be a lixiviant, or a reagent used for leaks, often in tanks, gold in solids and forms a cyanide structure of gold. Leaching takes put in tanks devoted for leaching taken after by adsorption onto carbon in tanks devoted for adsorption.

(Zagury, Oudjehani, & Deschênes, 2004).

Active carbons (ACs) are overcrowded building materials with a unique structure: well-designed microscope structure, wide internal space, long recurrence distance, dynamic advertising power, and flexible advertising. In the gold hydrometallurgy industry, activated carbon is used for gold adsorption in the form of AuCN _2 particles that kicks inside the cyanide filter mixers with a minimum volume of 10 mg of gold. Within the gold industry, two AC-based advances are broadly utilized, and within the world, 80% of the gold generation utilizes one of these strategies. (Tsang et al., 2007).

Activated carbons adsorption capability can be exploited by a variety of methods. Adjusted pore structure and adjustable working groups on a given carbon surface determine the performance of active carbon. Recently, the commercial potential of carbon dioxide activated in the system of thiosulphate has been improved with the conversion of cupric ferrocyanide or silver ferrocyanide, or (MBT). (Shafeeyan, Daud, Houshmand, Shamiri, & Pyrolysis, 2010).

For recovery of gold, gold ore was taken and added thiosulfate solution in a pyrex beaker placed on a magnetic stirrer for 120 minutes at 150 degrees temperature. The next step is to recover gold from the thiosulphate solution. 0.3 g prepared column of activated carbon is used for 1 hour. The recovery method will be adsorption and gold get recovered on the surface of activated carbon. These dissolution experiments are carried out for AA analysis. The purpose of this study is to investigate different factors that influence the adsorption of gold and to investigate the drawbacks of cyanide leaching.

2. HYDROMETALLURGY

It is a process in which precious metals are extracted from ores by using different aqueous solutions as leaching solutions. It involves two main steps:

1. Leaching or dissolution
2. Recovery of metal

Hydrometallurgy could be a procedure inside the field of extractive metallurgy, the getting of metals from their minerals. Hydrometallurgy includes the utilization of aqueous solutions for the recuperation of metals from metals, concentrates, and reused or leftover materials. The treatment of gold-carrying materials can be done by using:

2.1 Leaching or Dissolution

The primary method of extracting gold from ores is cyanidation. Hard-to-handle materials are those in which extraction of gold is difficult and have required examination of other extraction methods for substances. less expensive substances are chloride, thiosulfate, sulfide, ammonia, and sulfites, the initial two are a lot of significant for wellbeing and security issues and have a low ecological effect. Taking all things together the above specialists, thiosulfate has a lot of significance, as it can extract gold and can form a gold thiosulphate complex. Precious metals in WPCBs obtain by pyrometallurgy, hydrometallurgy, and electrometallurgy because toxic compounds are released. The techniques of hydrometallurgy (leaching, solvent extraction, ion exchange, etc.) are used for the recovery of metal. It is a favorable experiment. A large number of effluents are generated by hydrometallurgical treatment, if discharged into the environment, they can cause harmful diseases. (Ventura, Futuro, Pinho, Almeida, & Dias, 2018). Therefore, only thiosulphate solutions are used as leaching solutions to dissolve gold from gold-bearing ore.

The leaching of ammoniacal thiosulfate plays a potential role as a protective and non-toxic process (than cyanide) for the separation of gold in different ores. On the other hand, we faced different problems like the lack of an effective method for gold recovery. It has its main applications in the extraction of chlorine on paper and in-house textiles, mechanical repairs, soap storage, and chemical regent. Thiosulfate has many applications to treat skin worms such as worms. For centuries, ammonium thiosulfate has been used as a sulfur soil fertilizer. In nature, there are different advantages to cyanide because it is low toxic and environmentally friendly. Because high convergences of ammonium thiosulfate in water cause the development of green growth in waterways and lakes. (Meyer, DeAngelis, & Heineman, 1977).

The thiosulphate solution is preferred because of:

- It could provide far faster recovery than cyanide in mining exploiting “preg-robbing” ore deposits.
- Since thiosulfate leaching is a hydroxide mechanism (generally in the pH range of 8 to 10), there are also no issues with product corrosion mostly during the process of recovery.
- The copper-catalyzed thiosulfate leaching process has many advantages over the traditional cyanidation process.
- Thiosulfate draining of complicated and carbonaceous-type minerals can be ordered as a non-harmful cycle; gold disintegration rates can be more prominent than past cyanidation, and high gold recuperations can be acquired from thiosulfate filtering and carbonaceous-type metals because of lower obstruction from unfamiliar cations.

2.2 Recovery of metal

For the recovery of gold from the gold complex, a 0.3 g prepared column of activated carbon is used for 1 hour. The recovery method will be adsorption and gold get recovered on the surface of activated carbon. The adsorption detachment innovation is viewed as the most effective technique since it is exceptionally proficient and harmless to the ecosystem. Numerous adsorbents have been concentrated to get metal particles, for example, cell sorbent, zeolites, and activated coal. Nonetheless, these things have restricted publicizing limits or are costly. In this way, analysts have been endeavoring to build up a compelling and reasonable adsorbent. (Xiang et al., 2020).

Gold adsorption by using activated carbon in ammoniacal solution is studied here. Some factors that depend upon the adsorption of gold are temperature, concentration, pH, etc. The reaction has activation energy which is 19.4 KJ/mol. Initially, the rate of adsorption increases but decreases at the end. pH value increases as the rate of adsorption also increases. When the concentration of gold increases, the rate also decreased but when the concentration of thiosulphate increases, the initial rate also increased but in the end rate decreases. The concentration of ammonia also increases and its rate of adsorption also increases. Other factors also take part in the increasing rate of gold adsorption. To increase the rate of gold adsorption, we prefer the ammonium solution. As the rate of gold adsorption increases, more gold is extracted from gold-bearing ore. The optimum pH will be 9.5 and activation energy will be 19 KJ/mol. If we required gold in a greater amount, used a different type of solution. This recovered gold will be used in different fields. Gold is used in different fields i.e., in aerospace, medical field, coinage making, and in jewelry making. It is used worldwide in making jewelry almost 73 percent of total production. In Pakistan, most deposits of gold are found in Baluchistan. (Hammer & Norskov, 1995).

In a leaching solution, we also compare the rate of gold dissolution. The rate of cyanide dissolution in carbonaceous gold ores from gold strike deposits was less than in thiosulphate solution because of the preg-robbing phenomenon. Cyanide has a greater attraction with preg-robbed carbonaceous ores leading to the removal of gold from the gold cyanide complex back and resulting in poor dissolution. Due to the low attraction of carbonaceous materials for gold thiosulfate complex, this process does not occur through thiosulfate leaching. This also suggests that active carbon adsorption, which is the most popular process for recovering pregnant cyanide solution, would be ineffective in recovering the dissolved gold in thiosulfate solution. The rate of dissolution of cyanide in non-carbonaceous ores was greater than in the thiosulphate solution. In short, thiosulphate solution has less rate of dissolution than cyanide.

Extraction of gold by a cyanide solution	Extraction of gold from ammoniacal thiosulphate solution
A greater amount of gold is recovered by using cyanide because the gold cyanide complex has a strong affinity with activated carbon sites.	Less amount of gold is recovered from the gold thiosulphate complex. Because this complex has less affinity with active sites of carbon.
Its strong affinity will be due to its small size and less charge of gold cyanide complex. Therefore, it has a high rate of recovery or adsorption.	Less affinity will be due to the large size and large charge of the gold thiosulphate complex. Therefore, it has less rate of adsorption than cyanide.

TABLE 1: Compares the rate of gold adsorption or recovery on AC (activated carbon)

3. MATERIALS, METHODS, AND EXPERIMENTAL WORK

3.1 Materials

3.1.1 Chemicals

Activated carbon and ammoniacal thiosulphate solutions are purchased from the chemical lab center in Anarkali Bazar Lahore. Activated carbon has a length of 5mm, a diameter of 3 mm, and a surface area of 930 m^2. The micropores of 96% ammonium hydroxide solution and reagent-grade chemicals are used.

3.1.2 Apparatus & Equipment

The following apparatus is used for both experimental techniques which consist of roasting, leaching, and analysis.

- 1ml pipettes for delivery of less than 1ml reagent
- 5ml and 10 ml pipettes for the introduction of reagent up to 5 and 10 ml.
- Graduated cylinder (25ml & 100ml)
- Measuring flasks (25ml &100ml)
- Beaker (125ml)
- Graduated test tube (25ml)
- A test tube stands with simple test tubes
- Vacuum desiccator
- Glass reactor (500ml)
- Distill and deionized water

3.1.3 Sample Collection

Sample ore is taken from Dr. Mustansar, former Head of Geology, PU Lahore.

3.2 Methodology

Step I Dissolution:

0.1 gm ore was taken and added 100 ml thiosulfate solution of 5% concentration in a pyrex beaker placed on a magnetic stirrer for 120 minutes at 150 degrees temperature. The next step is to recover gold from the thiosulphate solution.

Step II Recovery:

For recovery of gold from the gold complex, 0.3 g prepared column of activated carbon is used for 1 hour. The recovery method will be adsorption and gold get recovered on the surface of activated carbon.

3.3 Experimental work

Solution preparation for characterization:

Solution preparation:

Recovery of gold is done by using activated carbon from a solution of ammoniacal thiosulphate, a stock solution of gold needs to prepare for characterization.

Standard Stock Solution Preparation:

0. 1M gold solution in 100 ml prepared by weighing 0.1 grams of gold and adding it into 100 ml flask. Add aqua regia in it as a standard solution to make it up to 100 ml.

Working Solution:

- Take a stock solution of different concentrations of 10, 20, 40, 80, 200, 400 ppm and add water to it and find absorption against known concentration. This dissolution is used for AA analysis. Then find the absorption of a sample before and after adding activated carbon.
- Absorption of standards and ore solutions are checked by the AA. Graph of absorption vs concentration in ppm is obtained.
- These solutions are also subjected to activated carbon for adsorption for 1 hour and 8 hours to find percentage recovery.

Analyze % Recovery of gold:

- The test of gold recovery is proceeded out in a glass reactor of 500ml reactor bath having a shaking mechanism, with constant temperature in the water bath. Different experiments have different concentrations of gold sample solution + ammoniacal thiosulphate solution at a 500 min^{-1} stirring point at different temperatures of 10,….,200 degrees temperature and reaction time is 15,..., 200 minutes. Gold dissolves in thiosulphate solution and a graph of different concentrations of gold vs absorption is obtained. The unknown concentration of gold before adding activated carbon is known and represented by C_0.
- Then a prepared column of activated carbon is dipped in the above solution of concentration C_0. Then gold from the solution gets adsorbed over the surface of activated carbon. Now, the concentration of gold C_t (after adding AC) in that solution needs to be known. Place this solution in AAs, then concentration after activated carbon is known. Now it will be less than concentration C_0.
- The concentration of unknown is found by calibrating the curve. Follow Bear Lambert's law:

$$A = \varepsilon c l$$

A	Absorbance	
ε	Molar absorption coefficient	$M^{-1}cm^{-1}$
c	Molar concentration	M
l	optical path length	cm

Figure 1: Bear Lambert's law

A= 0.6 nm, C of unknown will be 6ppm before adding activated carbon $\longrightarrow C_0$

A= 0.4 nm, C of the unknown will be 4 ppm after adding activated carbon $\longrightarrow C_t$

$$A= (C_0 - C_t) / C_0 \times 100$$

By putting values, % recovery of gold is found: % recovery will be 33.33 %. Only 33.33 % of gold is recovered.

Concentration (ppm)	Absorption (nm)
0	0
10	0.4
20	0.6
30	0.8
40	1
50	1.2
60	1.4
70	1.6
80	1.8
90	2

TABLE 2

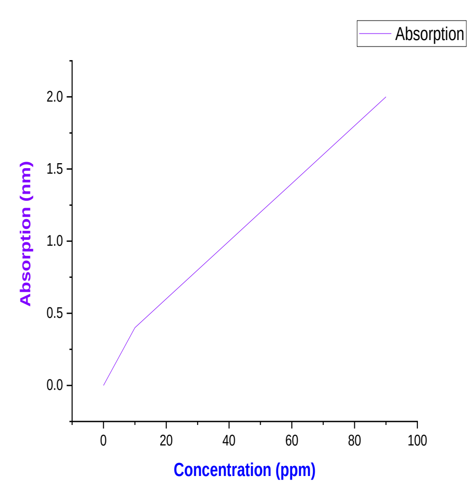

Figure 2: Graph showing the relation between concentration (ppm) and absorption (nm)

Reaction Mechanism Followed:

In an aqueous phase, gold forms a strong complex with thiosulphate, as seen in Eq I.

$$Au + 2S_2O_3 \rightarrow Au(S_2O_3)^{-2} \quad \text{[Eq. I]}$$

Eq II depicts the complex redox reaction in an alkaline thiosulphate solution with oxygen as an oxidant.

$$4Au + 8S_2O_3 + O_2 + 2H_2O \rightarrow 4Au(S_2O_3)^{-2} + 4OH \text{ [Eq. II]}$$

Due to its slow rate of thiosulphate gold leaching in oxygen, a proper catalyst is needed. Copper (II) is a highly accurate catalyst that keeps copper (II) in solution as the Cu (II) tetra-amine complex when combined with ammonia, as shown in Eq III.

$$Cu^{2+} + 4NH_3 \rightarrow Cu(NH_3)^{+2} \quad \text{[Eq. III]}$$

Gold produces stable complexes not only in thiosulphate solutions but also in ammonia solutions.

$$Au(S_2O_3) + 2NH_3 \rightarrow Au(NH_3)^{+2} + 2(S_2O_3)^{-2} \quad \text{[Eq. IV]}$$

In an ammoniacal copper-thiosulphate solution, Eq V depicts the complete gold dissolution reaction:

$$Au + 5S_2O_3 + Cu(NH_3)^{+2} \rightarrow Au(S_2O_3)^{-2} + 4NH_3 + Cu(S_2O_3)^{-5} \quad \text{[Eq. V]}$$

Cu (II) should be stable as the Cu (II) tetraamine ion in the presence of ammonia in the solution.

4. FACTORS DEPEND UPON ADSORPTION OR RECOVERY OF GOLD

Some factors that depend upon the adsorption or recovery of gold are temperature, concentration, pH, etc. which are discussed here:

4.1 Effect of ammonium hydroxide:

The results of these experiments show that at shorter times, the adsorption of gold on the carbon increased with increasing ammonium hydroxide concentration up to 0.6 mol/l and then remained constant, while the effect of ammonium hydroxide concentration is less significant at longer reaction times, at which the percentage of gold adsorbed onto the carbon is practically the same at the three different ammonium hydroxide concentrations used. This result may be attributed to the competitive adsorption of ammonium ion with gold ion on the carbon surface, i.e., an increase in ammonium hydroxide concentration results in a decrease in activity and diffusivity of gold ion, leading to a decrease in gold adsorption.

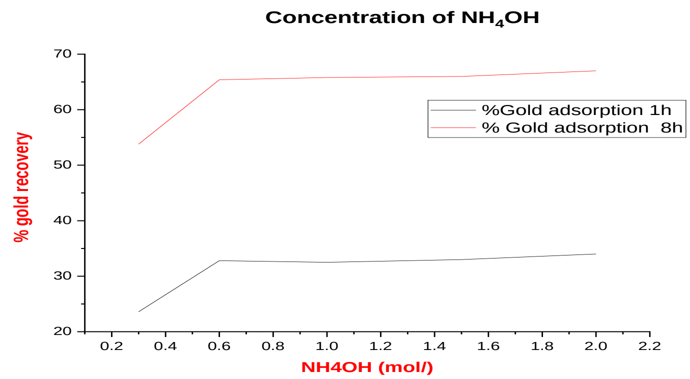

Figure 3: Graph showing the concentration of NH_4OH on % of gold recovery

4.2 Effect of pH:

As can be seen from the results shown in Fig 4, the pH variation did affect the gold adsorption, i.e., it is severely retarded at the lower and higher pH values, with optimum gold adsorption taking place at the pH of 10.5. As pH increases, the rate of adsorption also increases. This effect is dominant at the start but at the end of the reaction, further increases in pH lead to a decrease in adsorption behavior.

pH	% Gold adsorption (1 h)	% Gold adsorption (8h)
8.5	11.3	26
9.5	18.5	29.5
10.5	31.5	55.4
11	4.4	23.3

TABLE 3: Effect of pH on & gold recovery

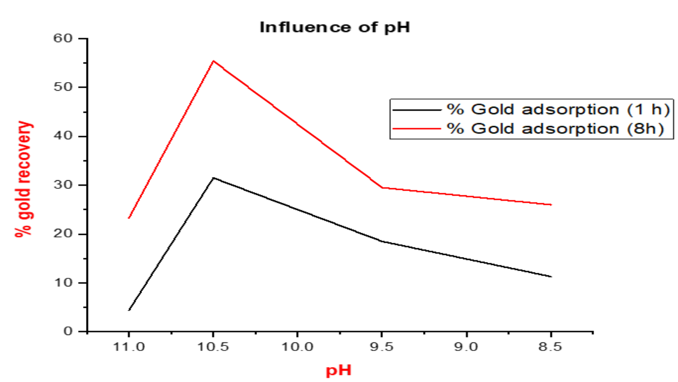

Figure 4: Graph showing the effect of pH on % gold recovery

4.3 Effect of temperature:

Figure 5 shows the effect of temperature on the adsorption of gold. It is seen that, within the range of temperature investigated, the adsorption of gold on activated carbon increased with increasing temperature. The activation energy was estimated at 19.4 kJ/mol.

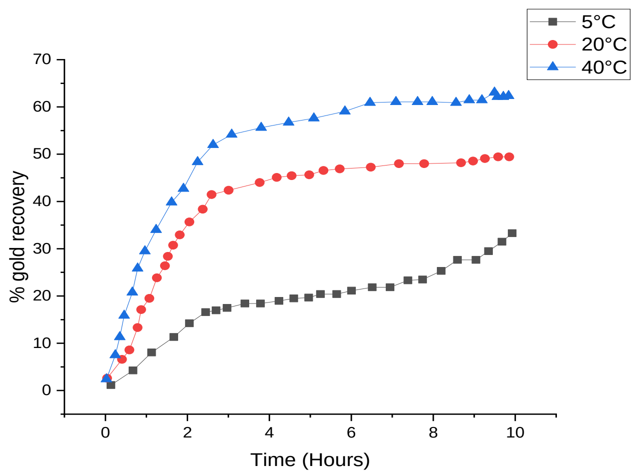

Figure 5: Graph showing the effect of temperature on % gold recovery

4.4 Effect of thiosulfate concentration:

Figure 6 plots the percentage of gold adsorption vs time for experiments performed with various initial thiosulfate concentrations. It can be seen that the presence of this anion in the aqueous solution influences the adsorption of gold onto the activated carbon. Two effects can be seen: first, the adsorption rate increases with increasing initial thiosulfate concentration, and second, the percentage of gold adsorbed during a given length of time decreases with increasing thiosulfate concentration. This is a serious drawback because practical leaching is expected to contain an excess of thiosulfate.

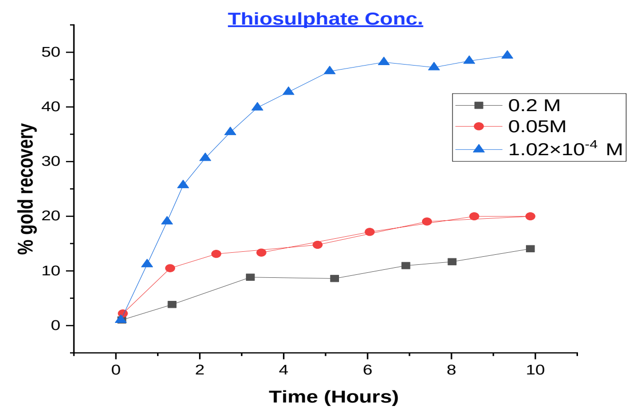

Figure 6: Graph showing the effect of thiosulfate concentration on % gold recovery

4.5 Effect of gold concentration:

As can be seen from Figure 7, the initial gold concentration has a significant influence on the adsorption of gold. It is clear that the higher the initial concentration of gold, the lower the adsorption rate. Moreover, the percentage of gold adsorbed onto the carbon increased as the initial gold concentration decreased. This result can be understood by assuming that the available active sites are insufficient to increase gold loading proportionately to the increase of gold in the initial aqueous solution, thus decreasing the percentage of gold adsorption onto the carbon.

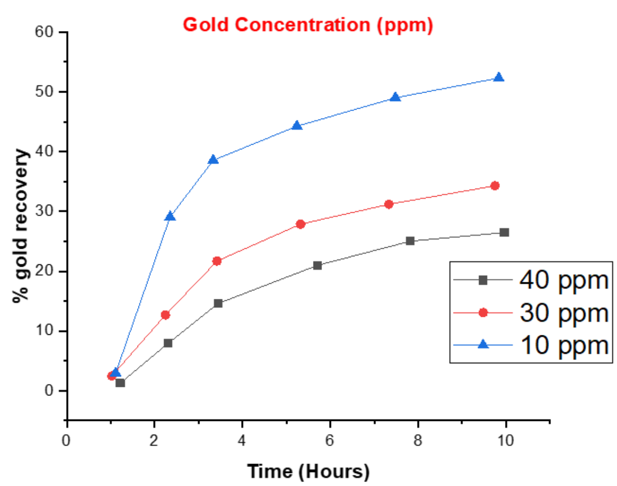

Figure 7: Graph showing the effect of gold concentration on % gold recovery

4.6 Effect of activated carbon:

In figure 8, it can be seen that the recovery of gold increases with a decrease in the above ratio, while the adsorption rate is only slightly affected by the variation of the amount of carbon added to the solution. It can be deduced that the surface area of the carbon plays an important role in the adsorption, because the more carbon used, the greater the surface area. Typical metal loadings of the carbon after the reaction time of 8 h were found to be 2g, 6g, and 3 g carbon, respectively for the solution volume to weight of carbon ratios of 1600, 800, and 480 (ml solution/g carbon), respectively.

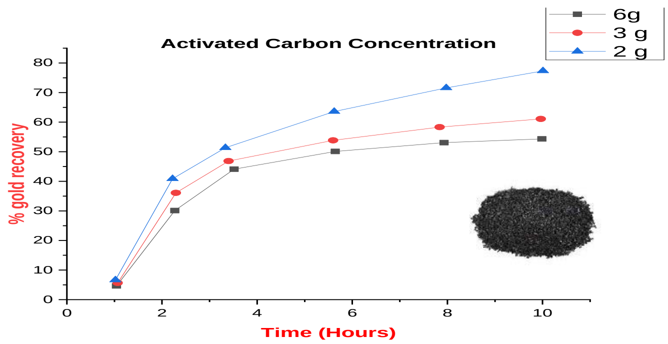

Figure 8: Graph showing the effect of activated carbon % gold recovery

5. CONCLUSION

Gold has been recovered from the ammoniacal solution by activated carbon. Different solutions to gold have been investigated. Different parameters are also observed. The influence of temperature, pH, and concentration of gold has been investigated. An increase in ammonium hydroxide concentration results in a decrease in activity and diffusivity of gold ions, leading to a decrease in gold adsorption. An increase in pH leads to a decrease in adsorption behavior. The adsorption of gold on activated carbon increased with increasing temperature. The activation energy is estimated at 19.4 kJ/mol. The adsorption rate increases with increasing initial thiosulfate concentration. The decrease in gold adsorption in the presence of these anions is also attributable to a decreased diffusivity of gold species caused by the anions. The higher the initial concentration of gold, the lower the absorption rate. As the concentration of copper increased, the percentage of gold recovered became less. Only 33.3% of gold has been recovered.

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